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10/733998
SQ0007USNA

RESPONSE AND REPLACEMENT BRIEF ON APPEAL

APPENDIX A - CLAIMS APPENDIX
CLAIMS (Pages A-1 to A-5)

APPENDIX B - EVIDENCE APPENDIX
WO01/88962
US2002/0071951A1

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AUG 30 2007 PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE
BEFORE THE BOARD OF PATENT APPEALS AND INTERFERENCES

In the Application of:

KURIAN ET AL.

CASE NO.: CH2825 US NA

APPLICATION NO.: 10/434,038

GROUP ART UNIT: 1723

FILED: MAY 8, 2003

EXAMINER: J.W. DRODGE

FOR: TREATMENT OF NON-AQUEOUS ALDEHYDE WASTE STREAMS

RESPONSE AND REPLACEMENT BRIEF ON APPEALCommissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

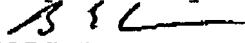
Sir:

The Appellants hereby submit, in response to the "Notice of Non-Compliant Appeal Brief" dated 14 August 2007, a "Replacement Brief on Appeal" addressing the issues raised in the Notice.

While the Appellants can find no basis in the rules or MPEP for the objection made in Paragraph 4 of the Notice, as explained in Paragraph 10 of the Notice, the Appellants have altered Section VI of the Brief to reference each element of independent claims 1 and 16 separately to the specification by page and line number.

The Appellants, therefore, request that this appeal proceed on the basis of the attached Replacement Brief on Appeal.

Respectfully submitted,


BART E. LERMAN
ATTORNEY FOR APPELLANTS
REGISTRATION NO. 31,897
TELEPHONE: 302-992-5285
FACSIMILE: 302-992-5374DATE: 8/30/2007

PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE
BEFORE THE BOARD OF PATENT APPEALS AND INTERFERENCES

In the Application of:

JING C. CHANG ET AL.

CASE NO.: SO0007 US NA

APPLICATION NO.: 10/733,998

GROUP ART UNIT: 1732

FILED: DECEMBER 10, 2003

EXAMINER: L.B. TENTONI

FOR: PROCESSES OF MAKING STAPLE FIBERS (AMENDED)

Commissioner for Patents
P.O. Box 1450
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REPLACEMENT BRIEF ON APPEAL

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I. INTRODUCTION

This is an appeal from the final rejection of claims 1-30 entered in the Final Office Action dated 6 November 2006.

A Notice of Appeal against the final rejections was filed 7 February 2007. As a consequence, a Petition for an Extension of Time under 37 C.F.R. §1.136, requesting a two (2) month extension of time, is also enclosed.

II. REAL PARTY IN INTEREST

The real party in interest is E. I. du Pont de Nemours and Company (Wilmington, DE).

III. RELATED APPEALS OR INTERFERENCES

It is believed that there are no related appeals or interferences that will directly affect or be directly affected by or have a bearing on the Board's decision in this pending appeal.

IV. STATUS OF THE CLAIMS

This application was originally filed with claims 1-45, directed to a first process for producing a 6-25 dpf carpet staple fiber (claims 1-15); a second process for producing a 1-6 dpf textile staple fiber (claims 16-30); various fibers produced by the aforementioned processes (claims 31-37 and 44-45); and various products made with such fibers (claims 38-43).

In a First Office Action dated 8 June 2006, a restriction requirement was entered requiring the Appellants to elect between the process claims (Group 1, claims 1-30) and the fiber and fiber article claims (Group 2, claims 31-43). In this First Office Action, the title of the application was objected to, and claims 1-30 were also rejected on prior art grounds.

In an "Amendment and Response" dated 29 August 2006, the Appellants elected to prosecute the Group 1 claims, and canceled the Group 2 claims without prejudice on any sort. The Appellants further amended the title of the application to its current form, as well as

claims 1, 6, 7, 15, 16, 22, 23 and 30, and responded to the rejections of record.

In the aforementioned Final Office Action dated 6 November 2006, one rejection was withdrawn but the rejections currently on appeal were maintained against all of claims 1-30.

The Appellants filed a "Response After Final Rejection" dated 5 December 2006, but the rejections of record were again maintained in an Advisory Action mailed 8 January 2007.

The claims remaining in this application and involved in the present appeal are thus claims 1-30. A clean copy of all claims in this application (including their status) is attached hereto as Appendix A.

V. STATUS OF AMENDMENTS

No amendment to the claims was filed subsequent to the Final Rejection, and no amendment remains outstanding.

VI. SUMMARY OF THE CLAIMED SUBJECT MATTER

The present application is directed generally to processes for preparing two different types of staple fiber from poly(trimethylene terephthalate) ("PTT"):

finer textile staple fibers of 1-6 dpf (claims 16-30), and

thicker carpet staple fibers of 6-25 dpf (claims 1-15).

Specifically, the invention of claim 1 is a process for producing 6 to 25 dpf carpet staple fiber (original claim 1, and lines 17-20 on page 4) comprising the steps of:

melt spinning poly(trimethylene terephthalate) into fibers (line 24-25 on page 11);

accumulating the fibers under conditions to produce an aged undrawn yarn (lines 16-27 on page 7, and "1" in Fig. 1);

prewetting the aged undrawn yarn, said aged undrawn yarn consisting essentially of poly(trimethylene terephthalate), at a tem-

perature less than about 45°C (lines 24-30 on page 11, and "2" in Fig. 1);

drawing the yarn under wet conditions at a temperature of from about 45°C to about 95°C in a first stage to a length of about 30 to about 90 percent of its final length (lines 1-28 on page 12, and "9" in Fig. 1);

further drawing the yarn in a second stage at a temperature from about 45°C to about 98°C under wet conditions (lines 5-14 on page 13, and "18-21" in Fig. 1);

crimping the drawn yarn (lines 16-28 on page 15, and "27" in Fig. 1);

thermo-fixing the crimped yarn in the presence of steam at a temperature from about 80°C to about 100°C (lines 6-10 on page 16, and "28" in Fig. 1); and

drying the crimped yarn at 60°C to 140°C (lines 11-17 on page 16, and "29" in Fig. 1).

The invention of claim 16 is a process for producing 1 to 6 dpf textile staple fiber (original claim 16, and lines 17-20 on page 4) comprising the steps of:

melt spinning poly(trimethylene terephthalate) into fibers (line 24-25 on page 11);

accumulating the fibers under conditions to produce an aged undrawn yarn (lines 16-27 on page 7, and "1" in Fig. 1);

prewetting the aged undrawn yarn, said aged undrawn yarn consisting essentially of poly(trimethylene terephthalate), at a temperature less than about 45°C (lines 24-30 on page 11, and "2" in Fig. 1);

drawing the yarn under wet conditions at a temperature of from about 45°C to about 95°C in a first stage to a length of about 30 to about 90 percent of its final length (lines 1-28 on page 12, and "9" in Fig. 1);

further drawing the yarn in a second stage at a temperature from about 45°C to about 98°C under wet conditions (lines 5-14 on page 13, and "18-21" in Fig. 1);

crimping the drawn yarn (lines 16-28 on page 15, and "27" in Fig. 1);

thermo-fixing the crimped yarn in the presence of steam at a temperature from about 80°C to about 100°C (lines 6-10 on page 16, and "28" in Fig. 1); and

drying the crimped yarn at 60°C to 140°C (lines 11-17 on page 16, and "29" in Fig. 1).

Preferred claimed embodiments applicable to both processes include:

(i) both the prewetting and drawing being carried out under water or under an aqueous solution of processing finish (lines 20-24 on page 12);

(ii) the first draw stage being carried out at a temperature of about 80°C or less, more preferably about 70°C or less, still more preferably about 60°C or less, and still more preferably about 50°C to about 55°C (lines 2-7 on page 12);

(iii) the second draw stage being carried out at a temperature of about 60°C to about 80°C (lines 9-11 on page 13);

(iv) the thermofixing being carried out at a temperature of about 85°C (original claims 13 and 29); and

(v) the crimped yarn being dried at a temperature of from about 60°C to about 100°C (lines 13-16 on page 16).

Preferred claimed embodiments specific to the process for producing carpet staple fibers include:

(i) the undrawn yarn being spun on equipment having a spinneret capillary density of at least 2/cm² and a quench zone shorter than about 16 feet (paragraph bridging pages 14-15 and lines 10-11 on page 15);

(ii) the undrawn yarn being spun at a speed less than about 600 ypm (lines 25-28 on page 14);

(iii) during the prewetting and drawing, the yarn being in the form of a spun rope of less than about 300,000 denier/inch (lines 29-32 on page 12);

(iv) in said first draw stage, the yarn being drawn to a length from about 40 to about 70 percent, more preferably from about 50 to about 55 percent, of its final length (lines 11-18 on page 12); and

(v) the drawn yarn having a denier of 6 to 20 dpf (original claim 14).

Preferred claimed embodiments specific to the process for producing textile staple fibers include:

(i) the undrawn yarn being spun on equipment having a spinneret capillary density of at least 8/cm² and a quench zone shorter than about 16 feet (paragraph bridging pages 14-15 and lines 10-11 on page 15);

(ii) the undrawn yarn being spun at a speed of 1300 ypm or less, and more preferably 900 ypm or less (original claims 18 and 19);

(iii) during the prewetting and drawing, the yarn being in the form of a spun rope of less than about 200,000 denier/inch (lines 29-32 on page 12); and

(iv) in said first draw stage, the yarn being drawn to a length from about 40 to about 90 percent, more preferably from about 70 to about 90 percent, of its final length (lines 11-18 on page 12).

As discussed throughout the present application, the problem addressed by the invention is how to overcome processing problems on a commercial scale when drawing aged undrawn PTT fibers (see. e.g., lines 18-28 on page 2, and lines 16-27 on page 7, of the specification). Commercial scale PTT processes normally result in aged PTT fibers because of the processing time to collect sufficient tow cans

(see "1" in Fig. 1) of PTT fiber for running through the drawing steps. Aging causes the PTT fibers to shrink and become brittle.

There are known ways to address this shrinking (aging) issue. For example, instead of ultimately collecting the spun PTT in tow cans, it is known to collect the fibers on takeup reels or similar devices, and to feed the so-collected PTT fibers directly into the drawing process. In this case the fibers are maintained in an effectively tensioned state, thus not allowed to significantly relax (and shrink), and thus would not be aged undrawn yarn within the meaning of the present invention. While useful, this means for controlling the problem is not practical at commercial spinning speeds and under other commercial conditions.

Additional measures to control the shrinkage and aging are disclosed, for example, in Casey et al (WO01/68962 discussed in detail below), which describes cooling the spun fibers at some point (e.g., during the quenching process) prior to collection on takeup reels (under tension), then preferably maintaining the collected fibers in a cooled, climate controlled room (see, e.g., the disclosure in Casey et al at lines 11-18 on page 3, and line 26 on page 10 though line 10 on page 11). Prior to removing the fibers from the cooled conditions, Casey et al recommends to make sure the undrawn yarn is under uniform roll tension, with preconditioning prior to drawing occurring under tension as well. Again, the predrawn yarns treated as in Casey et al would not be aged undrawn yarn within the meaning of the present invention.

While the process as disclosed in Casey et al is useful, all of the additional conditions for controlling the aging problem are not practical at commercial spinning speeds and under other commercial conditions.

Casey et al does indicated (lines 7-10 on page 11) that, "[e]ven if the PTT UDY shrinks it is possible to convert this UDY into a first grade commercial staple product during draw processing with minor impact on product quality". Casey et al, however, does not appear to teach specifically how to convert the shrunken (aged) PTT undrawn yarn into first grade product. In other words, Casey et al does not disclose what process conditions are necessary to con-

trol/adjust in order to draw aged undrawn PTT yarn. That is exactly the problem being addressed by the present invention!

VII. GROUND OF REJECTION TO BE REVIEWED ON APPEAL

A. 35 U.S.C. §102(b) - are claims 16-30 anticipated by the disclosure of Casey et al (WO01/68962)?

B. 35 U.S.C. §103(a) - are claims 16-30 unpatentable over the disclosure of Casey et al.?

C. 35 U.S.C. §103(a) - are claims 1-15 unpatentable over the disclosure of Casey et al, as applied to claims 16-30, and further in view of Hernandez et al (US2002/0071951A1)?

VIII. ARGUMENT

A. ANTICIPATION REJECTION

As just indicated, one of the grounds to be reviewed on appeal relates to the rejection of claims 16-30 as allegedly anticipated by the disclosure of Casey et al. The Appellants respectfully traverse this rejection.

The invention of claims 16-30 is, on a broad basis (paraphrased), a process for producing a 1-6 dpf textile staple fiber comprising the following steps:

- (1) melt spinning a PTT fiber;
- (2) accumulating the melt spun fibers under conditions so as to result in an aged undrawn yarn;
- (3) prewetting the undrawn aged PTT yarn at a temperature of less than about 45°C;
- (4) drawing the prewetted PTT yarn under wet conditions at a temperature of from about 45°C to about 95°C in a first stage to a length of about 30 to about 90 percent of its final length;
- (3) further drawing the partially drawn PTT yarn in a second stage at a temperature from about 45°C to about 98°C under wet conditions;

(4) crimping the drawn yarn;

(5) thermo-fixing the crimped yarn in the presence of steam at a temperature from about 80°C to about 100°C; and

(6) drying the crimped yarn at 60°C to 140°C.

The present invention, when considered as a whole, provides a combination of process conditions and steps that the Applicants have found effective to solve the problem identified above. Admittedly, Casey et al generally discloses a number of these conditions and steps, but it is the claimed combination and totality of these conditions, steps and circumstances which must be evaluated for patentability.

It is well established that anticipation requires the disclosure in a single prior art reference of each and every element of a claimed invention arranged as set forth in the claim. See, e.g., Connell et al. v. Sears, Roebuck & Co., 220 U.S.P.Q. 193, 198 (Fed. Cir. 1983). It is thus not sufficient for anticipation purposes for a prior art reference to simply disclose the various elements - the disclosure must show those elements arranged in a manner identical to the arrangement set forth in the claims. The Appellants submit that the disclosure of Casey et al fails to meet this standard.

As applied to the anticipation rejection based on Casey et al, the Applicants would specifically note that, as indicated above, it Casey et al does not even appear to disclose processing conditions specifically applied to aged undrawn PTT fibers as required by the present claims. At best, the disclosure of Casey et al is certainly ambiguous on this aspect.

As discussed in detail previously, Casey et al describes cooling the spun fibers at some point (e.g., during the quenching process) prior to collection on takeup reels (under tension), then preferably maintaining the collected fibers in a cooled, climate controlled room (see, e.g., the disclosure in Casey et al at lines 11-18 on page 3, and line 26 on page 10 though line 10 on page 11). Prior to removing the fibers from the cooled conditions, Casey et al recommends to make sure the undrawn yarn is under uniform roll tension,

with preconditioning prior to drawing occurring under tension as well.

Aged undrawn yarn in the context of the present invention is not on a takeup reel under tension as described in Casey et al. In fact, all the above-mentioned steps taken by Casey et al are specifically to avoid the creation of aged undrawn yarn as that term is understood in the context of the present invention. As indicated previously, while useful, all of the additional conditions used in Casey et al for controlling the aging problem are not practical at commercial spinning speeds and under other commercial conditions.

The Examiner does not address this gap in the disclosure of Casey et al, but instead focuses on the drawing process disclosed in the reference. Even in this limited view, the Appellants submit that the disclosure of Casey et al does not meet the conditions required to anticipate the subject matter of claims 16-30.

In addition to the prewetting step, the claimed process requires that both the first and second drawing stages be conducted under wet conditions as discussed in the present specification in, for example, the paragraph bridging pages 10-11 (and Figure 1), in lines 19-28 on page 12; lines 5-9 on page 13; and lines 10-20 on page 14.

Among other differences, Casey et al does not unambiguously disclose that both drawing steps (when more than one drawing step is used) are conducted under "wet conditions" within the meaning of the presently claimed invention. While Casey et al does seem to disclose a prewetting step (last paragraph on page 11), with respect to the draw steps Casey et al only discloses that "the initial draw point of the UDY tow should occur under water..." (emphasis added). This seems to correspond to a "wet" first stage within the context of the present invention, but a discussion of the wetting for the second stage as required by the present claims is totally absent.

The Examiner's view is that Casey et al does in fact disclose a second stage drawing under wet conditions, referring to page 12, lines 12-23, of Casey et al. The Appellants respectfully submit that the Examiner has taken this disclosure of Casey et al out of context.

Specifically, the Appellants would point to the following statement of Casey et al from the passage cited by the Examiner (at lines 16-19):

"If desired, the second draw stage is hotter than the first draw stage up to a practical maximum of the melting point of the yarn, preferably 60 to 160°C...."

It is not understood how, at the melting point of the yarn (or at 160°C), the second stage draw could unambiguously occur under wet conditions as, for example, a water bath would boil at those temperatures.

Casey et al, therefore, does not disclose (and certainly does not unambiguously disclose) that both drawing steps (when more than one drawing step is used) are conducted under "wet conditions" within the meaning of the presently claimed invention.

Taking all of this into consideration, it is evident that the Casey et al does not disclose the invention as set forth in claim 16 (and other claims dependent thereon), and does not anticipate the claims of the present application for the purposes of 35 U.S.C. §102(b). The Appellants, therefore, respectfully request reversal of this rejection.

With respect to dependent claims 17-30, the Appellants would note that, while these claims are explicitly rejected under 35 U.S.C. §102(b), the Examiner has provided no facts in support thereof. The Examiner has not even provided a basis as to where the limitations of these claims may be found in the disclosure of Casey et al. As such, the Appellants submit that the Examiner has not established a *prima facie* case of anticipation of these claims, and request that the rejection thereof under 35 U.S.C. §102(b) on the current record be reversed as well.

B. FIRST OBVIOUSNESS REJECTION

The Appellants traverse this rejection of claims 16-30 as allegedly being obvious solely on the disclosure of Casey et al.

Admittedly, Casey et al does disclose individually many of the steps of claims 16-30, but also discloses many variations of the same

steps that would clearly be outside the present claim scope. As discussed above, Casey et al also appears to fail to disclose applying any of these steps to aged undrawn PTT yarn within the meaning of the present claims.

In any event, in view of the Supreme Court's recent statements in the KSR case (KSR International Co. v. Teleflex Inc., 550 U.S. ___, 2007 WL 1237837, No. 04-1350 slip op. (2007)), the Appellants submit that the determinative question in deciding the appropriateness of this rejection is whether or not the Appellants claimed combination of steps yields predictable results with respect to being able to draw aged undrawn PTT yarn as required by the present claims. See also Leapfrog Enterprises Inc. v. Fisher Price, Inc. and Mattel, Inc., No. 06-1402 slip op. at 7 (Fed. Cir. 2007).

As discussed previously, Casey et al provides extensive direction on how to avoid aged undrawn PTT yarn (within the meaning of the present invention), but does not seem to provide coherent direction on how to deal with this problem should it occur. Dealing with this problem is precisely what the present invention is all about.

The Appellants respectfully contend that there is nothing in Casey et al or the current record to remotely suggest that the selection of steps, as embodied in the present claims, would predictably result a process that could be used to draw aged undrawn PTT yarn, as required by the present claims. There is simply nothing of record to support selecting steps and operating conditions that may be explicitly disclosed in Casey et al, or modify those steps or operating conditions not explicitly disclosed in Casey et al, in the manner required to achieve the presently claimed invention.

"[A] patent composed of several elements is not proved obvious merely by demonstrating that each of its elements was, independently, known in the prior art." KSR, No. 04-1350 slip op. at 14. Further, the mere fact that the prior art could be modified does not make the modification obvious unless the prior art suggests the desirability of such. See In re Gordon, 221 U.S.P.Q. 1125, 1127 (Fed. Cir. 1984).

Even reaching beyond the specific problem addressed by the Appellants, there is no other evidence of record that any need or prob-

lem known in the field of endeavor and addressed by this application would provide a reason for combining the various elements in the manner claimed. Without such evidence, the obviousness rejection cannot stand. See KSR, No. 04-1350 slip op. at 16.

In fact, the only possible way to arrive at the presently claimed invention from the fair disclosure of Casey et al, or the present record as a whole, is with hindsight benefit of the Appellants' disclosure and claims. Hindsight, however, is an inappropriate perspective in which to judge patentability. In re Deminski, 230 U.S.P.Q. 313, 316 (Fed. Cir. 1986).

In view of the above, the Appellants submit that, on the present record, (i) the Examiner has not established a *prima facie* case of obviousness of claims 16-30 based on the disclosure of Casey et al standing alone, and (ii) these claims are in fact patentable over any supportable reading of Casey et al.

The Appellants, therefore, respectfully request reversal of this obviousness rejection as applied to claims 16-30 (and the present claims as a whole).

C. SECOND OBVIOUSNESS REJECTION

The Appellants also traverse the second obviousness rejection of claims 1-15 as allegedly being obvious over the disclosure of Casey et al in view of Hernandez et al.

As set forth by the Examiner in the Final Office Action, Hernandez et al is cited solely for the proposition that PTT fibers having a dpf of 6-25 are known in the literature. The Appellants submit this fact has absolutely no bearing on the present question of obviousness in view of the established differences between the disclosure of Casey et al and the presently claimed invention.

Hernandez et al does not, in fact, deal with the issue of drawing aged undrawn PTT yarn, and does not otherwise explicitly or implicitly provide guidance to a person of ordinary skill in the relevant art how to modify the disclosure of Casey et al in order to achieve the presently claimed solution. In fact, the Appellants sub-

mit that no supportable combination of Casey et al with Hernandez et al even adds up to the presently claimed invention.

As with the first obviousness rejection, the only possible way to arrive at the presently claimed invention from the fair disclosures of the cited art is with the impermissible hindsight benefit of the Appellants' disclosure and claims.

The Appellants, therefore, submit that, on the present record, the Examiner has not established a *prima facie* case of obviousness of claims 1-15, and that these claims are in fact patentable over any supportable combination of Casey et al with Hernandez et al.

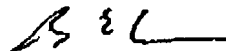
As a result, the Appellants respectfully request reversal of this obviousness rejection as applied to claims 1-15 (and the present claims as a whole).

IX. CONCLUSION

As is clear from the above as well as the arguments already of record, the approach taken by the Examiner and the rejections which flow therefrom clearly have no basis in law or fact. The Appellants, therefore, submit that the presently claimed invention is patentable over the art of record, request that the rejections of record be reversed, and further request that the Examiner be directed to:

- (i) allow claims 1-30, and
- (ii) advance the present application to issue at the earliest possible date.

Respectfully submitted,



BART E. LERMAN
ATTORNEY FOR APPELLANTS
REGISTRATION NO. 31,897
TELEPHONE: 302-992-5285
FACSIMILE: 302-992-5374

DATE: 8/30/2007

APPENDIX A - CLAIMS APPENDIX

Claims 1-43 from Application Serial No. 10/733,998

1. (Previously Presented) A process for producing 6 to 25 dpf carpet staple fiber comprising the steps of: melt spinning poly(trimethylene terephthalate) into fibers; accumulating the fibers under conditions to produce an aged undrawn yarn; prewetting the aged undrawn yarn, said aged undrawn yarn consisting essentially of poly(trimethylene terephthalate), at a temperature less than about 45°C; drawing the yarn under wet conditions at a temperature of from about 45°C to about 95°C in a first stage to a length of about 30 to about 90 percent of its final length; further drawing the yarn in a second stage at a temperature from about 45°C to about 98°C under wet conditions; crimping the drawn yarn; thermo-fixing the crimped yarn in the presence of steam at a temperature from about 80°C to about 100°C; and drying the crimped yarn at 60°C to 140°C.

2. (Original) The process of claim 1, wherein said undrawn yarn is spun on equipment having a spinneret capillary density of at least 2/cm² and a quench zone shorter than about 16 feet.

3. (Original) The process of claim 1, wherein the undrawn yarn is spun at a speed less than about 600 ypm.

4. (Original) The process of claim 1, wherein said prewetting and drawing are carried out under water or under an aqueous solution of processing finish.

5. (Original) The process of claim 1, wherein during said prewetting and drawing, said yarn is in the form of a spun rope of less than about 300,000 denier/inch.

6. (Previously Presented) The process of claim 1, wherein in said first draw stage, the yarn is drawn to a length from about 40 to about 70 percent of its final length.

7. (Previously Presented) The process of claim 1, wherein in said first draw stage, the yarn is drawn to a length from about 50 to about 55 percent of its final length.

8. (Original) The process of claim 1, wherein said first draw stage is carried out at a temperature of about 80°C or less.

9. (Original) The process of claim 1, wherein said first draw stage is carried out at a temperature of about 70°C or less.

10. (Original) The process of claim 1, wherein said first draw stage is carried out at a temperature of about 60°C or less.

11. (Original) The process of claim 1, wherein said first draw stage is carried out at a temperature of about 50°C to about 55°C.

12. (Original) The process of claim 1, wherein said second draw stage is carried out at a temperature of about 60°C to about 80°C.

13. (Original) The process of claim 1 wherein said thermo-fixing is carried out at a temperature of about 85°C.

14. (Original) The process of claim 1 wherein said drawn yarn has a denier of 6 to 20 dpf.

15. (Previously Presented) The process of claim 1 wherein the crimped yarn is dried at a temperature from about 60°C to about 100°C.

16. (Previously Presented) A process for producing 1 to 6 dpf textile staple fiber comprising the steps of: melt spinning poly(trimethylene terephthalate) into fibers; accumulating the fibers under conditions to produce an aged undrawn yarn; prewetting the aged undrawn yarn, said aged undrawn yarn consisting essentially of poly(trimethylene terephthalate), at a temperature less than about 45°C; drawing the yarn under wet conditions at a temperature of from about 45°C to about 95°C in a first stage to a length of about 30 to about 90 percent of its final length; further drawing

the yarn in a second stage at a temperature from about 45°C to about 98°C under wet conditions; crimping the drawn yarn; thermo-fixing the crimped yarn in the presence of steam at a temperature from about 80°C to about 100°C; and drying the crimped yarn at 60°C to 140°C.

17.(Original) The process of claim 16, wherein said undrawn yarn is spun on equipment having a spinneret capillary density of at least about 8/cm² and a quench zone shorter than about 16 feet.

18.(Original) The process of claim 16 wherein said undrawn yarn is spun at a speed of 1300 ypm or less.

19.(Original) The process of claim 16 wherein said undrawn yarn is spun at a speed of 900 ypm or less.

20.(Original) The process of claim 16 wherein said prewetting and drawing are carried out under water or under an aqueous solution of processing finish.

21.(Original) The process of claim 16, wherein during said prewetting and drawing, said yarn is in the form of a spun rope of less than about 200,000 denier/inch.

22.(Previously Presented) The process of claim 16, wherein in said first draw stage, the yarn is drawn to a length from about 40 to about 90 percent of its final length.

23.(Previously Presented) The process of claim 16, wherein in said first draw stage, the yarn is drawn to a length from about 70 to about 90 percent of its final length.

24.(Original) The process of claim 16, wherein said first draw stage is carried out at a temperature of about 80°C or less.

25.(Original) The process of claim 16, wherein said first draw stage is carried out at a temperature of about 70°C or less.

26. (Original) The process of claim 16, wherein said first draw stage is carried out at a temperature of about 60°C or less.

27. (Original) The process of claim 16, wherein said first draw stage is carried out at a temperature of about 50°C to about 55°C.

28. (Original) The process of claim 16, wherein said second draw stage is carried out at a temperature of about 60°C to about 80°C.

29. (Original) The process of claim 16, wherein said thermo-fixing is carried out at a temperature of about 85°C.

30. (Previously Presented) The process of claim 16, wherein said crimped yarn is dried at a temperature from about 60°C to about 100°C.

31. (Canceled)

32. (Canceled)

33. (Canceled)

34. (Canceled)

35. (Canceled)

36. (Canceled)

37. (Canceled)

38. (Canceled)

39. (Canceled)

40. (Canceled)

41. (Canceled)

42. (Canceled)

43. (Canceled)

44. (Canceled)

45. (Canceled)

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- (71) Applicant (for all designated States except US): SHELL OIL COMPANY [US/US]; One Shell Plaza, P.O. Box 2463, Houston, TX 77252-2463 (US).
- (72) Inventors; and
- (73) Inventors/Applicants (for US only): CASEY, Paul, Karol [US/US]; 6126 Plantation Bay Drive, Katy, TX 77449 (US); DANGAYACH, Kailash [US/US]; 755 Windbreak Trail, Houston, TX 77079 (US); OLIVERI, Linda, Harvey [US/US]; 23042 Lasham Drive, Katy, TX 77450 (US); SHIFFLER, Donald, Albert [US/US]; 1700 Hardee Road, Kinston, NC 28504 (US).
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(54) Title: POLY(TRIMETHYLENE) TEREPHTHALATE TEXTILE STAPLE PRODUCTION

(57) Abstract: A process for making textile staple fibre from poly(trimethylene) terephthalate (PTT) which comprises: (a) melt extruding PTT polymer at 245 to 253 °C, (b) spinning the extruded PTT into yarn using at least one spinneret, (c) moving the spun yarn to a first takeup roll wherein the distance from the spinneret to the roll is from 16 to 20 feet, (d) cooling the spun yarn to less than 31 °C before it reaches the roll, (e) prior to the draw process, preconditioning the yarn under tension at a temperature of at least 60 °C, (f) drawing the yarn at a temperature of at least 60 °C, (g) allowing the drawn yarn to relax at a temperature of up to 190 °C, and (h) crimping the drawn yarn at a temperature of 70 to 120 °C, and decreasing the drawn yarn feed denier into the crimper by 10 to 60 percent by denier.

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POLY(TRIMETHYLENE) TEREPHTHALATE
TEXTILE STAPLE PRODUCTION

Field of the Invention

Poly(trimethylene terephthalate) polymer is a new polyester resin suitable for use in carpet, textile, and other thermoplastic resin applications. Poly(trimethylene terephthalate) (PTT) is chemically an aromatic polyester resin made by the polycondensation of 1,3-propanediol (PDO) and terephthalic acid. Production of textile staple from PTT is possible on a wide variety of commercial processing equipment.

10

Background of the Invention

PET synthetic fiber staple production is often decoupled two-stage process. The first stage involves extrusion of Undrawn Yarn, which is stored for draw processing in the second stage. There are two main types of draw processes used in staple production Draw-Relax and Draw Anneal. The fundamental difference between these two processes is how fiber shrinkage is managed. In draw relax staple production the shrinkage strategy is to pre-shrink the fibers in an oven after crimping to desired performance and properties. In draw annealed staple production the shrinkage strategy is heat the fibers allowing for constant length crystallization before crimping.

Staple fibre from polyethylene terephthalate (PET) has been produced and there are well established processes for doing so. It would be desirable to be able to produce PTT staple fibre on existing equipment. However, there are many differences between the two polymers which make the production of commercially useful staple fiber of PTT on existing staple production equipment difficult or unlikely. To understand how

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5 to produce PTT staple on existing equipment one needs to
address several process questions:

How do you characterize the draw behavior of undrawn yarn?
How does the draw behavior of undrawn yarn change over time? As
10 described in Example #1.

How do you control the properties of undrawn Yarn during
extrusion? As described in Example #2.

15 What is the typical draw performance for the undrawn yarn.
As described in Example #3 and Example #4.

How does one control fiber shrinkage during undrawn Yarn
production and storage as described in Example #5.

20

How do you control fiber shrinkage during the staple draw
and in the final staple products? As described in Example #6.

How does one crimp the fiber to provide texture and
cohesion in downstream commercial processes for staple textile
25 spun yarns and non-wovens? As described in Example #7

How do you heat set and control the youngs modulus and
stretch properties of the staple fiber. How do the staple
fiber properties impact the properties of spun yarns? As
30 described in Example #8.

What is a basic process to produce staple on existing
equipment that addresses the interdependent nature of the first
six process questions discussed above. Such a process is
35 provided by the present invention.

Summary of the Invention

The present invention describes a two-stage staple
40 production process using PTT. The first stage is extrusion of

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undrawn yarn (UDY). UDY is converted to a staple fibre product in the second draw production stage.

In accordance with the present invention there is provided
5 a process for making textile staple fibre from polytrimethylene
terephthalate (PTT) on existing PET textile staple fibre making
equipment which comprises: (a) melt extruding PTT polymer at
245 to 253° C, preferably 245 to 250 °C, (b) spinning the
extruded PTT into yarn using at least one spinneret, (c) moving
10 the spun yarn to a first takeup roll wherein the distance from
the spinneret to the first takeup roll is from 16 to 20 feet,
(d) cooling the spun yarn to less than 31°C, preferably less
than 25 C, more preferably less than 20 C, before it reaches
the first takeup roll, (e) optionally, storing the spun yarn in
15 a climate controlled room at a temperature of no more than 31°C
(both this step and the previous one are carried out to
minimize premature shrinkage of the undrawn yarn prior to draw
processing), (f) prior to the draw process, preconditioning the
yarn under tension at a temperature of at least 60°C,
20 preferably 60 to 100°C, (g) drawing the yarn at a temperature
of at least 60°C, preferably 60 to 100°C, with an optional
preferred second draw wherein the majority of the total draw
occurs in the first draw, most preferably 80 to 85% of the
total draw, and wherein the second and subsequent draws are
25 carried out at a temperature above the temperature of the first
draw up to a practical maximum of the melting point of the
yarn, preferably 60 to 160°C, most preferably at a temperature
of 80 to 100°C, (h) allowing the drawn yarn to relax at a
temperature of up to 190°C , preferably 100 to 140°C (the
30 relaxation can be from 2 to 25% or possibly more but is
preferably 2 to 10%) in order to achieve an increase in the
initial Young's modulus of the drawn yarn, and (i) crimping the
drawn yarn at a temperature of 70 to 120°C, preferably 80 to
120°C if the relaxation step is used and 70 to 100°C if it is
35 not used, and decreasing the drawn yarn feed denier into the
crimper by 10 to 60 percent by denier, preferably 40 to 60

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- 5 percent, from the drawn yarn feed rate used for making comparable PET staple in the existing equipment. Also, alternatively or in combination, the volume of the crimper may be increased 10 to 50, preferably 20 to 35 percent more than the crimper volume used to make PET in the existing equipment.
- 10 Preferably, the choice of conditions is based on the particular equipment and the desired yield.

Brief Description of the Drawings

- 15 The present invention will now be described by way of example with reference to the accompanying drawings, in which:

Figure 1 is a schematic of the process steps from resin to baled fibre whose critical elements will be described.

20

Figure 2 is the tenacity elongation balance curve useful in helping assess the possible range of staple properties for PTT.

- 25 Figure 3 shows a typical Stress / Strain Curve for as Spun Yarn Bundles.

Figure 4 shows the effect of extrusion temperature on fiber drawability.

30

Figure 5 shows the undrawn yarn shrinkage in water of different temperatures as a function of undrawn yarn spinning conditions.

- 35 Figure 6 shows the orientation schematic describing the effect of fiber shrinkage.

Figure 7 shows the effect of draw bath temperature and total orientation parameter on boil off shrinkage;

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Figure 8 shows the effect of draw bath temperature and total orientation parameter on 125°C dry heat shrinkage.

Figure 9 shows the effect of draw bath temperature and
5 total orientation parameter on 140°C dry heat shrinkage.

Figure 10 shows the effect of draw bath temperature and total orientation parameter on 175°C dry heat shrinkage.

10 Figure 11 shows the effect of draw bath temperature and total orientation parameter on 197°C dry heat shrinkage.

Figure 12 shows the effect of draw ratio and draw bath temperature on draw process relaxation factor.

15

Figure 13 shows the predicted dry heat shrinkage as a function of drier (relaxer) oven temperature for free relax 1.4 Total Orientation Parameter and 75°C draw bath temperature.

20 Figure 14 shows the effect of relaxer oven temperature and applied yarn stretch on 175°C dry heat shrinkage for 100% PTT yarns.

Figure 15 shows the effect of relaxer oven temperature and
25 applied yarn stretch on 175°C dry heat shrinkage for 100% PET yarns.

Figure 16 shows the comparison of PTT and PET spun yarn 175°C dry heat shrinkage at two yarn heat set temperatures.

30

Figure 17 shows the effect of relaxer oven temperature and applied yarn stretch on 175°C dry heat shrinkage for 50:50 PTT:Cotton yarns.

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Figure 18 shows the comparison of PTT and PET spun yarn boil off shrinkage at two heat set temperatures.

Figure 19 shows the comparison of PTT and PET spun yarn load at 5% strain at two yarn heat set temperatures.

Figure 20 shows the comparison of PTT and PET spun yarn 2 minute percent stress decay at two spun yarn heat set temperatures.

10

Figure 21 shows the comparison of PTT and PET spun yarn percent strain recovery (2 minute extension) at two spun yarn heat set temperatures.

15 Detailed Description of the Invention

Polymer textile staple is feasible using existing facilities. Since the equipment used by different companies varies greatly, there will be differences in how the processes are carried out. Once the staple producer tailors its settings to the unique properties of PTT, it is possible to produce a wide variety of staple products suitable for use in spun yarn and non-woven textiles. Textile staple produced from PTT offers excellent loft and drape providing softness, bulk, compatibility in blends, easy-care, and shape retention in textile products.

1.0 POLYMER MELTING

1.1 Resin Transfer and Drying

Low energy air conveying systems minimize dust formation when transferring resin from shipping containers, processing equipment and storage facilities. Before extrusion, PTT resin should be dried to a constant moisture level of 50 ppm or less. This moisture specification minimizes the impact of resin degradation by hydrolysis during melt spinning. Many types of commercial dryers using desiccated air have successfully met

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this requirement. Dryers equipped with molecular sieves (13X & 4A), vacuum systems, and lithium chloride desiccants have met moisture requirements in commercial production. When possible, it is preferable to dry the polymer using 13X molecular sieve
5 desiccated air (dew point of -40°C or less) heated to 130°C with 4-6 hours of drying time. Using dry air when transferring dried resin from the dryer to the extruder is essential to minimize hydrolysis during melt spinning.

In large commercial dryers, drying PTT at rates that can
10 keep pace with extrusion throughput may be challenging. In this situation higher dryer temperatures may be needed. The PTT dryer air temperature should not exceed 165°C . Dryer residence time should not exceed 4 hours when using 165°C air.

15 1.2 Undrawn Yarn (UDY) Extrusion

A typical melt preparation system includes an extruder, spin beam, melt pump, and spin pack. The key is to establish a uniform, optimum polymer melt viscosity by minimizing melt process temperature and residence time. Commercial production
20 of PTT UDY with both twin screw and single screw extruders is straightforward. In twin screw extruders it may be necessary to reduce the extruder melt pressure by as much as 25-50% (from PET conditions) to avoid excessive shear degradation of the polymer melt. Commercial production of PTT UDY uses extruder
25 melt temperatures ranging from 245°C to 270°C . Care must be taken when producing PTT UDY at melt temperatures between 260°C to 270°C to avoid excessive degradation of polymer melt and subsequent UDY properties. The optimal staple extrusion melt temperature for PTT is 245 to 253°C , preferably 245 to 250°C .
30 Future PTT resins with lower intrinsic viscosity will likely require lower temperatures. Figure 4 shows that better drawability is obtained when the polymer is extruded at 250°C rather than at 240°C or 260°C .

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2.0 UNDRAWN YARN (UDY) SPINNING

2.1 Spin Beams, Pumps, and Packs

Trials using mono-component and bi-component extrusion systems to produce PTT staple UDY were successful. Spin pump volume and revolution control systems sized for PET usually meet the lower through put per position requirement for PTT staple. The filtration media should have minimum pore size of 30 microns. Often commercial spin packs use a minimum amount of filtration media. In the early stages of developing a PTT staple production process, it is preferable to use a standard filtration depth of medium/course sand (90/120 mesh). Evaluation of filament diameter uniformity will help determine if spin pack filtration or extrusion system melt pressure need optimization.

Staple extrusion systems are engineered for a specific range of resin viscosity, throughput, melt temperature, and residence time. In general, the hole-throughput needed to make PTT staple is usually 20-30% lower than that for PET products of comparable denier. This essentially increases the residence time for PTT extruded with PET staple production equipment. The increase in melt residence time can lead to degradation if melt temperatures are higher than 260°C. Transfer line and spin beam heating systems should equal the extruder outlet polymer temperature if possible.

2.2 Spinnerets

Spinneret selection depends on the target product denier and is determined by the limiting throughput per hole-minute for stable melt spinning. In general PTT staple can use standard PET spinneret designs for similar products. However, PTT staple generally requires smaller capillary diameters for low denier products when compared to PET staple production. PTT resins have an upper shear rate limit of 7500-9000

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reciprocal seconds for round cross sections depending on melt extrusion conditions.

Spinneret selection based on target staple product denier is shown in Table I.

Table I: Spinneret Selection Chart

| Fine Denier Range at 1100m/ min | 1.0-1.3 dpf | 1.30-1.75 dpf | 1.40-1.85 dpf |
|---------------------------------|----------------|------------------|------------------|
| Spinneret diameter (mm) | 0.23 | 0.25 | 0.35 |
| Spinneret l/d | 1.95 | 1.85 | 1.85-2.0 |
| Limiting Throughput per hole | 0.32 | 0.41 | 0.45 |

It is important that a long fibre culmination zone (the distance from the spinneret to the take up roll) be used. This means that the zone should be 16 to 20 feet rather than the standard 8 to 12 feet for PET. In process shrinkage of PTT UDY is relatively high so the process must allow the fibre to establish a stable molecular structure before all of the filaments are combined into one large draw process feed yarn. In the production of PET staple fibre, this is not a significant problem. PTT has a more elastic crystalline morphology so the longer fibre culmination link helps stabilize the yarn allowing a manufacture to avoid additional air conditioning costs.

2.3 Quench

Trials using cross flow and radial quenching systems were successful. Radial quenching systems with both inside-to-outside and outside-to-inside airflow have been used successfully. The fibre bundle should be quenched quickly and uniformly to prevent UDY shrinkage in the spun supply cans. Quench temperatures ranging from 8-35°C have been used, although temperatures of 8-25°C are preferred. In general, quench airflow rates are limited by operability of the UDY thread path. The number of filaments per position has ranged from 350 to 3500 filaments per position. In fine denier staple

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production it may be possible to have upwards of 6250 filaments per position with modern radial quenching systems. Optimization of quench systems involves determining which conditions offer the most operability and yield the highest percent elongation for the target UDY creel properties.

2.4 Spin Finish

In this specification all the coatings applied to the PTT fibre during staple extrusion and draw production are defined as spin finishes. Spin finishes are fibre coatings that provide lubrication, cohesion, and additive protection to the PTT fibre during staple production and down stream processing. Both multi-component phosphate and mineral oil based finishes have been used successfully in the production of PTT staple. Proven PET spin finish chemistries and application methods are satisfactory for initial PTT staple products. Spin finish formulations and application methods can then be changed based on customer feedback on staple processing.

2.5 Take-Up

Take up speeds ranging from 900 to 1250 metres per minute have been used for commercial PTT UDY production. On research equipment, take up speeds for UDY have ranged from 500 to 2250 metres per minute. It is useful to take a controlled relaxation between the take-up capstan rolls and sunflower wheel before piddling into the tow can. Cooling all the filaments in a single position to less than 25-30°C is necessary to minimize UDY shrinkage in the tow can.

3.0 TOW DRAWING AND FINISHING

3.1 UDY Storage

Under normal storage conditions, PTT UDY completes over 90% of its ageing process within 8 hours of extrusion. UDY draw properties stabilize within 24 hours and no significant change in draw properties is observed after 2-4 months of storage at

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constant temperature. PTT UDY has the potential to shrink more easily and at lower temperatures than PET UDY. Storage conditions warmer than 25-30°C should be avoided because they trigger UDY shrinkage. Ideally, the PTT UDY creel is stored in an air-conditioned environment to help avoid shrinkage. The exact temperature that triggers PTT UDY shrinkage depends on UDY extrusion, quench, take-up and storage conditions. Even if the PTT UDY shrinks it is possible to convert this UDY into a first grade commercial staple product during draw processing with minor impact on product quality.

3.2 Creel Size

The creel size for PTT staple is determined by the size of the production crimper. In general the creel size for PTT staple is roughly 60% of an equivalent PET staple product because of the higher bulk of PTT fibres. A 600,000 denier drawn tow will satisfactorily feed a 110 mm wide by 20 mm high crimper. This may change as crimper size increases and/or draw production rates increase above 100-130 metres per minute. Since most draw production lines have a maximum line speed of 250-300 m/min, increasing the volume of the crimping chamber is another way to improve draw line productivity.

3.3 Creel and Tow Preparation

Avoid heating the PTT UDY above 25°C until the UDY tow is under uniform roll tension. This will minimize the shrinkage of PTT feeding the draw process and maintain a uniform fibre tension at all points in the tow cross section during drawing. If uncontrolled, non-uniform UDY shrinkage is allowed, can to can orientation variation will limit draw process uniformity.

Prewet baths before drawing are preferred, but the temperature should not exceed 25°C unless driven and nipped rolls are provided to minimize tension into the draw feed section. If driven rolls are not available, the bath should be at the lowest uniform temperature possible.

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3.4 Drawing Process

PTT staple has been manufactured on draw-relax and draw-anneal process configurations. In the draw relax process the staple is heat treated and dried under zero tension to reduce shrinkage. This process produces a low modulus fibre suitable for PTT spun yarns and blending with low modulus fibres like wool and acrylic. The draw anneal process heat treats the tow on rolls under high tension and produces a higher modulus fibre more suitable for blending with minor amounts of rayon, cotton or other higher modulus fibre.

The initial draw point of the UDY tow in the first draw stage should occur under water heated to a minimum of 60°C, preferably 60 to 100°C. Keeping the draw point hot improves draw process performance by significantly reducing the impact of extrusion conditions on production draw ratios. If desired, the second draw stage is hotter than the first draw stage up to a practical maximum of the melting point of the yarn, preferably 60 to 160°C, most preferably 80-100°C. Unlike PET, PTT will not turn harsh in heated draw baths. Additional draw zones are optional and usually extend the total machine draw ratio slightly. The major draw ratio should be taken in the first stage.

Annealing or relaxing PTT staple tow with a 3% roll relaxation across a set of 100-130°C calendar rolls increases the initial modulus of the final PTT staple by 12-14%. This process produces a high modulus fibre suitable for PTT spun yarns and blending with high modulus fibres like cotton, rayon, and PET. Initial modulus increases about 4% for every 10°C from 130-150°C when the relaxation across the roll set is held at 3%. Annealing PTT tow above 150°C may require increasing the relaxation across the calendar rolls to avoid excessive filament breakage. Spin finish is often applied to make up for spin finish lost in draw processing using a dip bath or

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front/back kiss roll application just before the crimping stage.

3.5 Crimping

5 PTT tow bends very easily compared to PET tow given its low bending modulus. This low modulus also gives PTT excellent hand and softness. Additionally, PTT is much more bulky than PET. Low bending modulus and high bulk require the following changes in crimping conditions:

10

- 10 Dancer roll and crimper roll are reduced to provide more control of crimp geometry.
- 15 Feed tow denier must be decreased or crimper volume increased because of PTT's higher bulk. The increased bulkiness of PTT can be accounted for by either decreasing the amount of tow denier feeding to the crimper by most preferably 10 to 60 percent, preferably 40 to 60 percent, all by denier. Another way is to increase the volume of the crimper by 10 to 50 percent, preferably 20 to 35 percent, all by volume. Also, a combination of these two methods can be used.
- 20 Ideally, the crimper should be equipped with steam and spin finish injection to better control crimping chamber temperature.
- 25 It may be necessary to improve the precision of pressure and temperature control in the crimping chamber.

30 Crimp stability and take up improve significantly when the crimper chamber is at least 85°C and at 300 kPa (3 bar) of gate pressure. Crimp frequency may be higher and crimp amplitude lower than comparable PET staple. Crimp stability and take up improves as crimper temperature increases. The crimper should not be heated too much because, as crimp stability increases, 35 so does staple cohesion, which can increase defects in carding.

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3.6 Drying, Cutting, and Packaging

Relaxation (drying) of PTT staple in conventional belt ovens is straightforward. However, both crimp geometry and shrinkage characteristics change as the oven temperature is raised above the hottest temperature in the preceding draw process. In draw relax staple production both staple fibre and subsequent spun yarn dry heat shrinkage decrease as the dryer temperature is increased.

10 In draw anneal staple production the relaxation oven is used as a dryer. The airflow rates are relatively high and the air temperature relatively low (75-90°C) to facilitate tow drying. These conditions are not hot enough to allow staple fibre relaxation or crimp geometry change. Staple tow has been
15 cut in commercial production using both rotary and pacific converter type cutters without modification. Both gravity and air conveyed staple balers have packaged PTT staple in commercial trials.

20 4.0 GENERIC RECIPES FOR 1.7, 2.5 AND 3.33 DTEX (1.5, 2.25, AND 3 DPF) STAPLE

Draft recipes for three typical staple products are briefly outlined in the following table. Every staple production
25 facility is different. It will usually take 2-3 attempts on a commercial line to identify a commercial process for PTT staple. These recipes were developed on small commercial production equipment. They may change slightly as processes are scaled up to larger equipment and higher production rates. The UDY is
30 produced using a 253°C melt temperature at 1100 m/min. To attain these draw ratios requires uniform extrusion conditions such that the filament diameter coefficient of variation is between 3-5% in all spinning positions. Further these draw ratios are obtained in very well controlled, modern process
35 equipment. It is not unusual for older equipment to only attain 75-85% of these draw ratios.

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The recipes in table II also describe a high and low shrinkage recipe for each target staple denier. The amount of draw production shrinkage decreases significantly as the first stage draw bath temperature is raised above 60°C. Further the amount of draw production shrinkage decreases slightly as the draw ratio increases. Lastly draw production shrinkage in the crimper and dryer are further reduced when using calendar rolls to anneal the fibre. Commercial product draw production rates of 100-130 metres per minute and development draw rates as high as 225 m/min have been used. The commercial draw relax process typically uses a 70°C first draw and a 100°C second draw. The commercial annealed staple process typically uses a 70°C first draw, a 100°C second draw, and 130°C calendar rolls with a 0.95 relaxation.

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Table II: Draft Recipes for 1.5, 2.25, 3.0 dpt Staple

| | | | | | | |
|--|----------|----------|----------|----------|----------|----------|
| Undrawn Yarn dpt | 4.0 dTex | 3.2 dTex | 6.4 dTex | 6.8 dTex | 9.2 dTex | 9.7 dTex |
| 1 st Draw Ratio D ² /1 | 2.73 | 2.73 | 2.74 | 2.74 | 2.75 | 2.75 |
| 2 nd Draw Ratio D ² /2 | 1.10 | 1.10 | 1.15 | 1.15 | 1.20 | 1.20 |
| Machine Draw Ratio | 3.00 | 3.00 | 3.15 | 3.15 | 3.30 | 3.30 |
| Draw Production Relaxation in Crimper & Dryer | High | Low | High | Low | High | Low |
| | 20% | 16% | 18% | 14% | 16% | 12% |
| Final Staple Product | 1.7 dTex | 1.7 dTex | 2.5 dTex | 2.5 dTex | 3.3 dTex | 3.0 dTex |

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4.1 STAPLE PROPERTIES

This discussion of tensile properties assumes that the creel stock is drawn to within 90% of tow breakout. A typical draw relax PTT staple fibre will usually have a tenacity of 2.7-3.0 cN/dTex and an elongation between 80-90%. A typical commercial draw annealed staple will have 3.4-3.5 cN/dTex and an elongation between 60-65%. The tenacity elongation balance curve below (Figure 2) is useful in helping assess the possible range of staple properties for PTT. High tenacity, low elongation PTT staple is very challenging to produce due to rapid relaxation of PTT tow under crimping conditions. Single filament test results on R&D equipment indicate fibre tenacities as high as 4 cN/Tex with 45% elongation. One may be able to produce PTT staple with tenacities greater than 3.5 cN/Tex on a highly optimized draw anneal process. In this effort the control of crimping conditions will be critical.

Example 1: Controlling Shrinkage of Undrawn Yarn During Extrusion and Storage

Evaluation of the undrawn yarn shrinkage of PTT UDY spun at two different locations under a wide variety of different conditions. Test results indicate that it is best to store PTT UDY below 31°C to avoid excessive shrinkage greater than 2-3%, as shown in Figure 5. This chart shows the percentage shrinkage in undrawn yarn immersed in water baths at several temperatures - 30°C, 31°C, 32°C and 35°C. The spinning conditions covered a range of drawn product denier per filament (dpf) range from 0.8 to 4.5 dpf and the operating range of throughput and takeup speed for different developmental staple production lines one at location A and another at Location B. This chart also shows that PTT UDY shrinkage is more a function of spinning conditions than quench air temperature. Location A uses 25°C quench air and location B uses 16°C quench air.

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- 5 Excessive undrawn yarn shrinkage is undesirable because it can increase staple product variability if not properly controlled.

Example 2: Shrinkage of Staple Process Drawn PTT Ribbons

10 Summary and Conclusions:

Drawing in baths of 60°C or higher eliminates any effect of spin induced structure on as drawn fibre shrinkage.

- 15 Drawn fibre shrinkage decreases with increasing draw bath temperature, but the effect is fairly small at temperatures above 60°C. Shrinkage also decreases with increasing total orientation (Draw Ratio), but the effect is very minimal at the higher draw bath temperatures. When as drawn shrinkage is insensitive to spinning and drawing settings, crimping becomes
- 20 more stable, and the product less variable. Drawing at temperatures above 60°C is recommended and should provide stable crimping operation.

- 25 Predicted relaxation factors for crimping and drying/relaxing as a function of drier/relaxation temperature were prepared from the shrinkage data. Although the shape of the curve is correct and can be used for extrapolation of known data, the magnitude of the factor appears too high.

- 30 It appears unlikely that product shrinkage as a function of drier / relaxation temperature can be predicted from shrinkage data for PTT.

Introduction

- 35 The range of spinning conditions examined on the pilot draw line used herein were:

- 240 to 260°C block temperature
- 0.432 to 0.865 g/hole min (0.4 mm capillary)
- 1000 to 2000 m/min spin speed

- 40 These supplies were drawn at three draw ratios:

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- 5 • Breakout Draw Ratio (BODR) minus 0.1
 • BODR - 0.2
 • BODR - 0.4

This, coupled with the large variation in spun orientation, gives a large range of spun orientation.

- 10 Three staple draw bath temperatures were used:
 • 40°C
 • 55°C
 • 70°C

15 This was the maximum practical operable range for the equipment.

Drawn ribbons were completely characterized. The shrinkage results are analyzed below.

- 20 Unlike tenacity and elongation, which in PTT, PET, and other melt spun polymers, is primarily a function of total orientation, shrinkage is a much more sophisticated probe of fibre structure, and is affected by orientation and, more importantly, by crystallinity.

25 The objective of this part of the experiments is to answer the following shrinkage questions, which are presented in their order of importance to process design.

- 30 1. Do differences in spin yarn structure persist through drawing, or does the drawing process erase them? This has important implications in the design of spin processes and procedures.
- 35 2. How does the draw bath temperature affect shrinkage? Residual shrinkage after drawing is a major factor affecting how easily a fibre can be crimped. In general, fibres with high shrinkage crimp easily. If shrinkage is a strong function of draw bath temperature and orientation, the crimper must frequently be rebalanced to reflect changing draw conditions, and crimping is less uniform.

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- 5 3. How much shrinkage will occur when a fibre is drawn and crimped and how is it affected by spin and draw conditions? This information is used to calculate the relaxation factor in the spinning model.
- 10 4. What additional product shrinkage will exist at higher temperatures when fibre is free relaxed in an oven at a given temperature?

15 A model that has proven useful for PET has the following premises:

When heated above glass transition temperature, unless restrained, a fiber will shrink until all the amorphous area has deoriented.

20 As temperature is raised above glass transition, additional oriented amorphous areas appear as crystals melt. These newly created amorphous areas then deorient, providing additional shrinkage. So, in general, the higher the temperature, the higher the shrinkage.

25 Several definitions, which are somewhat circular in nature, are now required. Glass transition temperature is defined as that temperature at which unrestrained amorphous chains are free to deorient, as required by the second law of thermodynamics. Amorphous areas are those which are not
30 crystalline. Crystalline areas are those which do not deorient at this temperature. There are any number of ways to define crystalline regions in terms of TGA curves, X-ray behavior, density etc, and all give more or less different, but related, answers in terms of % crystallinity. For the purposes of this
35 discussion we will define crystals in terms of their ability to retain orientation at temperature.

Inherent in this model is the assumption that heat treating a rope results only in crystalline melting and fibre
40 deorientation. For PET yarns relaxed in processes with short

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dwelt times, this model works well. It works fairly well for staple processes involving dwell times of several minutes, but its validity for PTT was unknown before this work.

5 The length, orientation diagram in Figure 6 can be used to illustrate the process. This diagram represents what happens when you draw a fibre from its length at zero birefringence to the sample fibre length, l_1 . When this fibre is heated to temperature T_1 it loses length to l_1 because all the amorphous
10 areas deorientate and all crystals that are not stable to T_1 melt and also deorientate.

 The fibre does not deorientate completely, because there are still crystals that are stable at T_1 . When the temperature
15 is raised to T_2 , additional crystals melt, become amorphous, deorientate, and the length decreases further. One would think that it would be possible to shrink completely to zero birefringence draw ratio 1.0, but in practice there are some crystals that are stable to and beyond the melting point,
20 (which causes problems in PET polymerization). These do not deorient and so reorientation can never be complete.

 With the above introduction behind us, it is now possible to address the experimental questions.

25 Do differences in spin yarn structure persist through drawing?

 One would expect that shrinkage should be a strong function of the draw ratio, because that is what provides the orientation that is lost. With PET, this is true to some
30 degree, but the effect is grossly overshadowed by the fact that as orientation increases, so does the ability to form crystalline areas, so there are opposing effects of orientation increase. Also in PET, at elevated draw bath temperatures, all memory of spinning is erased, in so far as shrinkage is
35 concerned. Orientation can be approximated as the total

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orientation parameter (TOP) (Denier Draw Ratio / Natural Draw Ratio).

To calculate the natural draw ratio, it is necessary to determine the proper strain rate for the laboratory equipment and skills which would give reproducible results. A single tube spun at 40#/hr, 240°C, and 1500 m/min was tested at strain rates from 200 to 800 % per minute. Three replicate measurements were performed with the curves traced on a single graph. Results were quite reproducible at all strain rates indicating good laboratory skills and equipment. All determinations gave the characteristic curve illustrated in Figure 3. Natural draw ratio can be calculated easily from these plots as follows:

15

$$\text{NDR} = 1 + (S_n / 100) \quad (1)$$

where:

S_n is % Strain at the Natural Draw Strain

20

This is equivalent to the classic definition of NDR which is:

$$\text{NDR} = l_d / l_s \quad (2)$$

25

where:

l_d is the length at the natural draw point of inflection

l_s is the length of the spun sample.

30

The first step is to establish what variables are statistically significant in predicting shrinkage at a given temperature. The procedure was to use the stepwise forward and stepwise backward regression procedures in Sigma Stat 2.0 with F to enter >4.0 and P to reject <0.05.

35

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Results summarised in Table III indicate:

- Both procedures were in general agreement.
- 5 • At draw temperatures of 60°C or higher, spinning variables play no significant role in drawn fibre shrinkage.
- 10 • Total Orientation Parameter and, surprisingly, block temperature were weakly significant factors in shrinkage at the 45°C draw temperature.

It is concluded that draw temperatures of 60°C or higher should be used to wipe out any spinning effects on product shrinkage. This data supports the hypothesis that, like PET, spinning structure is wiped out if a draw temperature significantly above T_g is used. Regression equations for correlations with $r^2 > 0.5$ are reported below the Table. Note that there are no such for $T \geq 60^\circ\text{C}$.

20

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Table III
Regression Analysis of Ribbon Shrinkage

| Draw Bath Temperature, °C | Shrinkage | Forward Test | | Backward Test | |
|---------------------------|-----------|--------------|----------------|---------------|-----------------------|
| | | Variables | r ² | Variables | r ² |
| 45 | Boil Off | TOP, Tb | 0.666 | TOP, Tb | 0.666 ⁽¹¹⁾ |
| " | 125 °C | TOP, Tb | 0.695 | TOP, Tb | 0.695 ⁽¹²⁾ |
| " | 140 °C | TOP, Tb | 0.540 | TOP, Tb | 0.540 ⁽¹³⁾ |
| " | 175 °C | TOP, Tb | 0.489 | TOP, Tb | 0.441 |
| " | 197 °C | TOP | 0.126 | TOP | 0.126 |
| 60 | Boil Off | Qh | 0.190 | Qh | 0.153 |
| " | 125 °C | TOP | 0.188 | Qh | 0.131 |
| " | 140 °C | TOP | 0.241 | TOP | 0.241 |
| " | 175 °C | AVE | na | AVE | na |
| " | 197 °C | AVE | na | AVE | na |
| 75 | Boil Off | AVE | na | AVE | na |
| " | 125 °C | Qh, Sm | 0.273 | Qh, Sm | 0.273 |
| " | 140 °C | AVE | na | AVE | na |
| " | 175 °C | AVE | na | AVE | na |
| " | 197 °C | AVE | na | AVE | na |

TOP = Total Orientation Parameter = (Denier Draw Ratio/Natural Draw Ratio)

Tb = Block Temperature, °C

Qh = Hole Throughput, g/hole minute

Sm = Spinning Speed, metres/minute

AVE = All Independent Variables Eliminated

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5 How does the draw bath temperature affect shrinkage?
Figures 7 through 11 are plots of Boil Off Shrinkage, and Dry
Heat Shrinkage at 125, 140, 175, and 197°C, for the three draw
bath temperatures used. It is clear that there is a large
mechanism change between 45 and 60°C, the next higher
10 temperature tested. At temperatures greater than 60°C shrinkage
is nearly independent of orientation, and relatively
insensitive to bath temperature.

Higher bath temperatures do decrease shrinkage potential a
15 small amount, but not significantly in terms of crimping
potential or probable product performance.

Operation at temperatures of 60°C or higher is recommended
because crimper operation is greatly simplified. Crimper
20 adjustments under these conditions are not required except to
compensate for denier density into the crimper (denier/linear
crimper inch, dtex/linear crimper cm).

How does spinning and draw bath temperature affect the
25 relaxation factor? Figures 7 through 11, and the analysis of
variance, indicate that shrinkage potential of drawn rope is
independent of spinning conditions and only weakly dependent on
orientation, except for the lowest temperature examined, 45°C.

30 Figure 12 shows the relationship between oven temperature
and relaxation factor for drawn ropes. At draw bath
temperatures above 60°C a line through the top grouping of data
points should approximate the relaxation factor. This is a
good starting point, but the value may be too high (shrinkage
35 too low) because the shrinkage method uses a small weight on
the sample so it is not completely free to relax as it would in
a typical plant drier/relaxer.

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However, the curve should have the correct shape and therefore be good enough to extrapolate the temperature effect when more machine specific data is obtained.

- 5 Can relaxed product shrinkage be predicted from the ribbon shrinkage data?

Referring to Figure 3 and the simple shrinkage model for PET it is found that if a fibre at l_f is put in an oven at T_1 ,
10 it will lose amorphous orientation and some of the crystals will melt and deorientate and its length will decrease to length l_1 . Similarly, if a sample of length l_f is placed in an oven at T_2 , which is higher than T_1 , it will shrink more because more of the crystalline material will melt at the higher.
15 temperature and shrink to length l_2 .

Mathematically, the shrinkage may be expressed in the following way:

20 Let: $\Phi_1 = \% \text{ Shrinkage @ } T_1 / 100$ (1)

$$\Phi_2 = \% \text{ Shrinkage @ } T_2 / 100 \quad (2)$$

Then, from the definition of Dry Heat Shrinkage:

25 $\Phi_1 = (l_f - l_1) / l_f = 1 - l_1 / l_f$ (3)

$$\Phi_2 = 1 - l_2 / l_f \quad (4)$$

What would happen if the sample free relaxed at T_1 is tested it for shrinkage at T_2 ? If no crystalline growth or change
30 other than melting and deorientation takes place during the first shrinkage process, it should shrink to l_2 .

The first shrinkage, then, is what the fibre experiences in the relaxer, and the second shrinkage is the residual shrinkage

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in the relaxed product. To some degree PET follows these assumptions so it is possible to estimate relaxed product shrinkage from the dry heat shrinkage of drawn ribbons.

- 5 To do this what must be calculated is the shrinkage when the product shrunk at T_1 is shrunk a second time at T_2 . This can be done by the using the definition of shrinkage and Figure 3 as follows:

10
$$\Phi_{ps} = (\% \text{ shrinkage of sample shrunk at } T_1 \text{ when shrunk at } T_2) / 100 \quad (5)$$

$$\Phi_{ps} = (l_1 - l_2) / l_1 = 1 - l_2 / l_1 \quad (6)$$

- If equations 3 and 4 are used to eliminate l_1 and l_2 in terms of the measured dry heat shrinkages at the two different
15 temperatures this leads to:

$$\Phi_{ps} = 1 - (1 - \Phi_2) / (1 - \Phi_1) \quad (7)$$

- From this, and the measured shrinkages, the expected product shrinkage at T_2 after an oven relaxation at T_1 can be
20 calculated.

- Figure 13 is a plot of predicted shrinkage for the technical useful case of high orientation, and high bath temperature. It appears that the predicted fibre shrinkages
25 after a given oven relaxation are much too low. This indicates that significant crystalline change in addition to simple deorientation occurs in the drier/relaxer.

- Example 3: Evaluation of PTT Staple Fiber Heat Setting on the Properties on PTT Spun Yarn Properties under Heated Strain
30 Conditions: Yarns Evaluated included PTT, PTT/PET Blend, PTT/Cotton Blend, and PET (Table IV)

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Breakage of filaments during the extrusion of PTT synthetic fibres severely limits production productivity and product quality. PTT resins with IV in the range of 0.55-1.0 are preferred, more preferably those with IV range of 0.675-0.92, and most preferably those with IV range of 0.72-0.82. Producing PTT synthetic fibres with an intrinsic viscosity range of 0.72-0.82 helps improve synthetic fibre production operability and product quality without a significant reduction in final fibre properties.

10

Reducing the intrinsic viscosity of PTT helps:

1. Reduce the amount of change in the viscosity of the chip compared to the viscosity of the extruded fibre.
- 15 2. Improve the homogeneity of polymer melt in the spin pack. PTT resin with 0.92 IV requires more severe spin pack filtration systems to maintain marginal yields in extrusion.
- 20 3. Improve production operability by decreasing the number of broken filaments during production.
4. Allow one to run the product with cooler extrusion temperatures for extruded filaments having less than 2-denier per filament. PTT is known to degrade at melt extrusion temperature above 260°C. When producing fine denier synthetic filament (filaments with less than 2 dpf) using 0.92 IV resin one has to increase the melt extrusion temperature to reduce the melt viscosity enough to avoid excessive melt flow turbulence and melt degradation which cause filaments to break during extrusion.
- 25 5. Reduce the amount of shrinkage in the product fibre making it easier to draw process and/or wind on to stable yarn packages.
- 30
- 35

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5 Staple yarns made from PTT are surprisingly elastic -
having recoverable elasticity when extended up to 15-25% of the
original yarn length. This elasticity is also present in
staple yarns produced from intimate and non-intimate fibre
10 blends where PTT is the major fibre component by weight and/or
length. Further, this elasticity is recoverable after several
hundred cycles. The elasticity is sufficient enough to enhance
the shape retention characteristics of textile fabrics produced
from PTT staple yarns and blended staple yarns. Properly
15 constructed and finished fabrics that contain a majority of PTT
staple spun yarn (by weight of length percent) can have
surprisingly high elastic recovery in woven and knit fabrics
(tested with over 500 cycles by hand and 200 by instrument).
The present invention covers the formation of staple spun yarns
by conversion of staple into a twisted yarn structure by any
20 method. The spun yarn can be made by hand, spinning wheel,
ring-spinning, open-end spinning, air-jet spinning or other
types of staple to yarn conversion equipment.

25 Staple yarns made from cotton, wool, acrylic, PET are not
elastic. In order to produce staple yarns from these fibres
that are elastic the industry commonly has to add an elastic
continuous filament either internally to the yarn or fabric to
give the final textile product elastic properties. These
30 solutions are more expensive than a basic staple spun yarn made
from PTT. The value of the present invention is that people
with basic staple spun yarn technologies can produce an elastic
spun yarn of commercial value without investing in more
expensive core spun yarn equipment or incorporating elastic
continuous filaments into the fabric structures, which then
35 complicates how the fabric is dyed and finished.

An effort was made to briefly characterize how staple fiber
heat setting properties impacts the behavior of yarns made from
PTT staple and PTT staple blended with cotton, and PTT staple
40 blended with PET staple. As noted in Table IV, the PET and

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- 5 cotton blend yarn were prepared from several PTT staple
supplies produced with differing relaxer temperature and having
significantly different crimp. So, the effect of blending with
other fibers cannot be definitely ascertained. Caution should
therefore be used in inferring precise blend level effects from
10 these experiments. This experiment looks at each of the
example yarns in terms of shrinkage, modulus, stress decay and
recovery. These factors are generally independent and were
studied separately in this work.

15 Table IV: Fiber Properties

| Item Number | PTT5-CR1-2 | PTT5-CR1-3 | PTT-CR1-4 |
|----------------------------------|------------|--------------------------|-----------------------|
| Fiber Relaxation Oven Temp °C | 105 | 120 | 135 |
| Fiber Denier/Filament | 1.75 | 1.80 | 1.79 |
| Tenacity, cN/dtex | 3.52 | 3.49 | 3.24 |
| Elongation to Break, % | 48 | 45 | 44 |
| Load at 10% Strain, cN/dtex | 0.83 | 1.21 | 1.13 |
| Hand Crimp Index, % | 28 | 19 | 18.5 |
| Crimp/inch | 14.8 | 15.8 | 14.2 |
| Yarn Produced | 100% PTT | 50%PTT and 50% Cotton | 50% PTT and 50%PET |

Summary and ConclusionsFor Spun Yarn 175°C Dry Heat Shrinkage:

- 20 • Spun yarn dry heat shrinkage decreased with oven
temperature for all blends tested (100% PTT, 100% PET,
50/50 PTT/PET, and 50/50 PTT/Cotton).
- Dry heat shrinkage increases approximately $\frac{1}{2}\%$ for each %
applied stretch (or relaxation) for all blends tested.

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- 5
- PTT spun yarn shrinkage was 2 to 2 ½ % less than PET yarns.
 - PTT data closely followed the amorphous deorientation model for shrinkage.
- 10
- For Spun Yarn Boil off Shrinkage:
 - Boil off shrinkage decreased with oven temperature for all blends tested.
 - Boil off shrinkage increases about 0.4% for each 1% applied stretch for all blends tested.
- 15
- PTT spun yarns were about 1% lower than PET yarns in boil off shrinkage, because they were made by the draw relax process whereas the PET sample was draw anneal.

For Spun Yarn Load at 5% Strain (Stretch):

- 20
- Fabrics are perceived as "stretchy" when very little force is required to change their length a significant amount. In this set of trials we choose to characterize stretch by observing the force required to strain the fabric 5%. The major variable affecting all blends tested was applied stretch.
- 25
- Oven temperature was much less important, this evaluation indicates:
- PTT exhibited 3-4 times less force to elongate 5% (greater stretch) than PET.
 - The amount PTT's stretch decreases with applied stretch is 1/10th that of PET (0.01 gpd increase/1% stretch vs 0.1)
- 30
- Therefore applied stretch can be used for PTT to modify yarn properties without a large yarn stretch penalty.
- The stretch of 100% PTT yarn was relatively insensitive to heat set conditions.

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5 Spun Yarn Stress Decay

How much a yarn or fabric will recover after being subjected to a given strain for a given length of time depends on two factors:

- 10 1. How much stress decay occurs while the strain is maintained.
2. The amount of recovery after the strain is released.

These factors are generally independent and were studied separately in this work:

15

- PTT stress decay was independent of heat set temperature and decreased linearly with increasing applied stretch (0.5% reduction in stress decay / 1% applied stretch in heat setting).

20

- PET stress decay decreased with increasing oven temperature and linearly with increasing applied stretch. The applied stretch effect is considerably stronger than with PTT (-0.9% per % applied stretch).

25

- PTT and PET exhibited roughly the same amount of stress decay.

30

- PTT / PET blend yarns behaved about half way between the corresponding pure yarns with a -0.7% decrease per % applied stretch.
- PTT / Cotton blend yarn stress decay was independent of heat setting conditions.

35 Spun Yarn Recovery

Recovery for the PTT yarns from this sample set was much less than that observed for the pre-commercial yarn sample recently tested having 98% recovery. The cause for this may be

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- 5 the 100°C drier oven used in fiber processing. The principal heat set variable affecting recovery for all samples tested was applied stretch, with increasing applied stretch increasing recovery:
- PTT recovery increases 0.9% per 1% applied stretch
 - 10 • PTT generally had 5 to 10% higher recovery than PET
 - PTT / Cotton blend data was very erratic but exhibited the same general trends as the pure yarns.

Shrinkage Fundamentals

- 15 In a semi crystalline polymer fiber with significant orientation, the orientation resides in two areas, the crystalline areas, and the amorphous areas connecting the crystalline domains. There is usually a range of crystal sizes, and the orientation of crystalline regions can vary.
- 20 When a fiber is exposed to temperatures below the glass transition temperature, length change is very slow and is called creep. In general, useful textile fibers all have low rates of creep in the absence of load. When the fiber is heated to temperatures above T_g, the amorphous areas become
- 25 mobile and in the absence of a restraining force, deorient as closely as they can to the isotropic state (no preferential orientation). The isotropic state is favored by the second law of thermodynamics. This usually results in shrinkage, but in rare instances the crystalline regions are collapsed upon
- 30 themselves and there is "negative" orientation so the fiber grows. Such fibers are called self elongatable. It is not known if they can be made from PTT. The crystalline regions are not mobile, and do not deorient.
- 35 As one continues to increase the temperature of the fiber, smaller crystals melt and their domain becomes amorphous. They now deorient and additional shrinkage occurs. This is why shrinkage generally increases with temperature for a semi

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5 crystalline polymer. So there are two strategies for reducing fiber shrinkage:

1. Preshrink to the temperature at which you want the fiber to be stable

10

2. Crystallize under heat and tension at a temperature which yields crystals stable at the temperature at which you do not want the fiber to shrink.

15 For commodity PET fiber, where the issues are spinning and weaving efficiency and yarn strength, the second route is exclusively used, because as we will see later, preshrinking reduces fiber modulus. Specialty fibers, particularly for wool blends, use the first route because strength and modulus are
20 not important issues, but the better dyeability offered by route 1 is an advantage. At this juncture, it is not clear which is the better route for PTT.

If a fiber is stretched, its amorphous orientation
25 increases, and so does shrinkage. In PET untwisted continuous filament yarn there is often nearly a 1 to 1 correspondence (5% stretch increases shrinkage 5%).

So far we have been discussed single uncrimped fibers.
30 The situation for spun yarn is more complex because:

- Fibers are twisted at a helix angle which reduces the effect of fiber shrinkage on the yarn
- 35 • Fibers migrate from outside to the middle of the yarn
- Fibers can slip in the yarn
- The presence of blend fibers with different shrinkages can
40 change the shrinkage of the assembly.

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5 Even with these complexities, we can use the simple model to predict responses of a spun yarn to heat setting conditions:

10 1. If the yarn is heated and allowed to free relax, it should deorient and crystallinity should increase. Both factors decrease shrinkage. At a constant oven temperature, the shrinkage decrease should be linear with the relaxation. Shrinkage should decrease with increasing oven temperature.

15 2. If a yarn is heated and held at constant length there is no deorientation. Shrinkage decreases with increasing oven temperature as long as the yarn treatment oven temperature is higher than the fiber experienced during the free to relax step in fiber manufacture.

20 3. If a yarn is heated and stretched, orientation increases and therefore so should shrinkage. Shrinkage should increase linearly with applied stretch, and decrease with increasing oven temperature, providing that it is higher than that seen during fiber processing.

25 With this foundation in place, we are now ready to analyze the effect of heat setting conditions on yarn shrinkage.

30

175 °C Spun Yarn Dry Heat Shrinkage

As illustrated in Figure 14, PTT is a wonderful substrate that follows all the rules of the total orientation model.

35 At 0% applied stretch the control which was relaxed at 100°C has exactly the same shrinkage as the yarn treated at 100°C. When treated at higher temperatures, still at constant length, the shrinkage decreases.

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5 As length is changed by applied stretch, or shrinkage, the shrinkage increases linearly with applied stretch, and the slopes are roughly constant with oven temperature. At a given oven temperature 176°C dry heat shrinkage increases about 0.46% per 1% applied stretch.

10

PET behaves in a similar manner, Figure 15. Because it is an annealed, and not a relaxed fiber, shrinkage of the control yarn decreases with increasing oven temperature even at 100°C. Because it is high modulus, at high stretch and low oven temperature the fibers do not stretch, but slip in the yarns so the shrinkage increase is not so great as for PTT. For oven temperatures above 130°C PET dry heat shrinkage increases about 0.55% per 1% applied stretch. This is somewhat higher than PTT, but a good rule of thumb for both is $\frac{1}{2}$ % shrinkage increase for each % stretch.

20

Figure 16 compares PTT and PET yarn shrinkage for the highest and lowest heat set temperatures. PTT has about 2 to 2 $\frac{1}{2}$ % lower dry heat shrinkage than PET for equivalent oven conditions. As mentioned above both have roughly the same increase in shrinkage with applied stretch.

25

The PTT / Cotton blend is similar to the PET blend (Figure 17) with shrinkage increasing with decreased oven temperature and increasing applied stretch. The increase with applied stretch is fairly linear and shrinkage increases roughly 0.47 % per 1% applied stretch. Shrinkage of the cotton blends is approximately 1% less than the PET blend for similar conditions. For this sample set a good rule of thumb is that 1% applied stretch increases dry heat shrinkage $\frac{1}{2}$ %.

30

35

Spun Yarn Boil Off Shrinkage

Boil off shrinkage behaves in the same way as dry heat, except that the amorphous orientation available is that present

40

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in the fiber plus all crystals which melt between the glass transition and 100°C, so it is much less than the dry heat shrinkage. In some fibers where the plastization effect of water is high, this shrinkage can be considerable. Fibers
5 produce by a draw relax process with relaxation temperatures above 100°C generally have very low boil off shrinkages. Annealed fibers generally have relatively high boil off shrinkage because they are heated under tension and there is always amorphous orientation present.

10

These samples in general followed these principles. All blends behaved in a manner similar to dry heat shrinkage with shrinkage decreasing with increasing oven temperature, and increasing with applied stretch.

15

The PTT control yarn had a shrinkage of 2% even though the fibers which went into it were oven relaxed at 100 C. This indicates that some cold drawing occurred in processing, probably during carding. This is not unexpected, given PTT's
20 low modulus. PTT yarn shrinkage increased about 0.36% per 1% applied stretch. The control PET yarn had considerably higher shrinkage than PTT (4.5 vs 2%) because it is produced by the annealing process. Yarn shrinkage increased 0.47% per 1% applied stretch. In general PTT yarns have approximately 1%
25 less dry heat shrinkage than PET yarns given similar yarn heat setting conditions (Figure 18).

The PTT/PET blend yarns had a shrinkage increase of 0.44% per 1% applied stretch, and the PTT/Cotton yarn had an
30 increase of 0.417. A good rule of thumb for boil off shrinkage is that it increases about 0.4% per 1% applied stretch for all conditions tested.

For 100% PTT spun yarns, load at 5% strain is nearly
35 independent of oven temperature, and is linearly related to applied stretch increasing 0.01gpd per 1% increase in applied

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5 stretch. This means that yarn stretch decreases as the yarn is drawn during heat setting. Note for the PTT data that the control yarn, which was produced from fibers relaxed at 100100% has essentially the same load at 5% strain as that heat set at 0 stretch and 100°C.

10

PET yarns behaved in a similar manner. In this case there was a considerable change for the control yarn vs yarn heat set at 100°C and 0 applied stretch because the feed fibers were annealed, not relaxed. PET load at 5% elongation increases an
15 order of magnitude higher than PTT with applied stretch, 0.1 gpd/1% applied stretch.

Figure 19 compares the behavior of PTT and PET, and PTT's stretch advantage is strikingly apparent. Not only is the
20 force required for 5% strain a factor of 3 lower with no applied stretch, the response to applied stretch is much less, which means applied stretch can be used in heat setting PTT yarns without paying an excessive price in yarn stretch. Interestingly the highest applied stretch PTT item (7.5%)
25 requires 45% less force at 5% strain than a PET sample which is relaxed 7.5%.

Spun Yarn Stress Decay

30 How much strain a yarn or fabric will recover after being strained and held at constant length for a period of time depends on two factors:

How much stress decay occurs while the sample is held at
35 constant length. In the extreme, if all the stress is lost, the recovery will be zero.

How much strain is recovered after the test time period.

40 This spun yarn stress decay experiment involved straining

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5 the yarn 5%, then holding the spun yarn at length for 2 minutes and allowing the yarn to recover to zero stress. Stress decay was calculated manually from the test charts, and is somewhat less accurate than machine calculated numbers from computerized analysis methods. Stress decay and recovery are two separate
10 phenomena and are discussed separately.

Stress decay of 100% PTT yarns was independent of oven temperature and decreased linearly with applied stretch. While intuitively it is believed stress decay should increase with
15 applied stretch, the r^2 for this correlation is quite high. PTT stress decay decreases about 0.5% per % applied stretch. PET yarn behaved in a similar way although there is a definite oven temperature effect, and the decrease per unit applied stretch (0.9% decay / 1% applied stretch) is nearly double that
20 of PTT. Figure 20 compares PTT and PET stress decay. In general PET is higher at low applied stretch, and lower than PTT at high oven temperature and applied stretch. The behavior of the PTT / PET blend is half way between the two pure fibers with less oven temperature sensitivity and a 0.7% stress decay
25 decrease per 1% applied stretch. Stress decay for the PTT / Cotton blends was independent of heat setting conditions.

Spun Yarn Recovery

30 PTT recovery was not affected by oven temperature and increased linearly with increasing applied stretch (0.9 % recovery per 1% applied stretch). PET recovery increased marginally with increase in oven temperature but the principal effect was applied stretch. Its response is much more
35 pronounced than for PTT with an increase of 2.2 % recovery for each 1% applied stretch. As indicated in Figure 21, PTT generally has 5-10% higher recovery than PET except where PET has had high levels of applied stretch in a high temperature oven. The PTT / PET blend yarn responded like pure PTT with no
40 oven temperature dependence and a strong response to applies

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- 5 stretch (1.7% increase in recovery for each 1% applied stretch. PTT / Cotton blend results were erratic with both higher oven temper and higher applied stretch increasing recovery.

Property Trade Off in Spun Yarn Heat Setting

10

- One of the primary reasons for doing this work is to answer the question: "If heat setting the yarn to reduce its shrinkage then how much does stretch, recovery and stress decay deteriorate?". To answer this question for this data set it is
15 necessary to plot these variables against each other. Since all variables are dependent variables, the relationships hold true only for this data set and others in which the dependent variables were changed in the same manner as we did here. With this caveat let us observe the responses:

20

- For the stretch / dry heat shrinkage tradeoff, stretch increases (load at 5% strain decreases) as dry heat shrinkage is decreased. This is a favorable trade since there is no stretch penalty to be paid by reducing shrinkage by letting the
25 yarn shrink. Although the r^2 is quite low at 0.47, considering that every point is included, this data is probably a reliable trend guide. Load at 5% decreases 0.01 gpd for each 1% reduction in shrinkage.

30

- The recovery / dry heat shrinkage tradeoff is unfavorable. Recovery decreases 1.3% for each 1% reduction in dryheat shrinkage. r^2 for this data was a respectable 0.64.

- The stress decay / dry heat shrinkage tradeoff is
35 unfavorable with stress decay increasing 0.9% for each 1% reduction in dry heat shrinkage. It is believed this increase in stress decay is responsible for the reduction in recovery. In any case dry heat shrinkage should only be reduced the minimal amount required by the end user.

40

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Example 4: Lowering Resin PTT Resin IV from 0.92 to 0.82
Provides Improved Extrusion Reliability in PTT Staple
Production

5 Breakage of filaments during the extrusion of PTT
synthetic fibers severely limits production productivity and
product quality. Producing PTT synthetic fibers with an
intrinsic viscosity range of 0.72-0.82 helps improve synthetic
fiber production operability and product quality without a
10 significant reduction in final fiber properties.

Reducing the intrinsic viscosity of PTT helps reduce the
amount of change in the viscosity of the chip compared to the
viscosity of the extruded fiber. It also improves the
15 homogeneity of polymer melt in the spin pack. PTT resin with
0.92 IV requires more severe spin pack filtration systems to
maintain marginal yields in extrusion. It also helps improve
production operability by decreasing the number of broken
filaments during production. It allows one to run the product
20 with cooler extrusion temperatures for extruded filaments
having less than 2-denier per filament. PTT is known to
degrade at melt extrusion temperature above 260°C. When
producing fine denier synthetic filament (filaments with less
than 2 dpf) using 0.92 IV resin one has to increase the melt
25 extrusion temperature to reduce the melt viscosity enough to
avoid excessive melt flow turbulence and melt degradation that
results in which cause filaments to break during extrusion. It
also reduces the amount of shrinkage in the product fiber
making it easier to draw process and/or wind onto stable yarn
30 packages.

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C L A I M S

- 5 1. A process for making textile staple fibre from
polytrimethylene terephthalate (PTT) on existing PET textile
staple fibre making equipment which comprises:
- 10 (a) melt extruding PTT polymer at 245 to 253° C,
- (b) spinning the extruded PTT into yarn using at least one
spinneret,
- 15 (c) moving the spun yarn to a first takeup roll wherein
the distance from the spinneret to the first takeup
roll is from 16 to 20 feet,
- (d) cooling the spun yarn to less than 31°C before it
reaches the first takeup roll,
- 20 (e) optionally, storing the spun yarn at a temperature of
no more than 31°C,
- (f) prior to the draw process, preconditioning the yarn
25 under tension at a temperature of at least 60°C,
- (g) drawing the yarn at a temperature of at least 60°C,
- (h) optionally, allowing the drawn yarn to relax at a
30 temperature of up to 190°C ,and
- (i) crimping the drawn yarn at a temperature of 70 to 120°C
and decreasing the drawn yarn feed rate by 10 to 60
percent by denier from the drawn yarn feed rate used
35 for making comparable PET.
2. The process of claim 1 wherein the intrinsic viscosity of
the PTT is from 0.55 to 1.0.

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- 5 3. The process of claim 2 wherein the intrinsic viscosity of the PTT is from 0.72 to 0.82.
4. The process of claim 1, 2 or 3 wherein the spun yarn is cooled to less than 25°C before it reaches the first takeup
10 roll.
5. The process of claim 1 wherein the relaxation step is not used and the crimping temperature is 70 to 100°C.
- 15 6. The process of claim 1 wherein the relaxation step is used and the crimping temperature is 80 to 120°C.
7. The process of any one of the preceding claims wherein step (i) further comprises using a crimper volume of 10 to 50
20 percent more than the crimper volume of the crimper used to make comparable PET.
8. The process of any one of the preceding claims wherein the drawn yarn feed rate is decreased by 40 to 60 percent.
- 25 9. A process for making textile staple fibre from polytrimethylene terephthalate (PTT) on existing PET textile staple fibre making equipment which comprises:
- 30 (a) melt extruding PTT polymer at 245 to 253° C,
- (b) spinning the extruded PTT into yarn using at least one spinneret,
- 35 (c) moving the spun yarn to a first takeup roll wherein the distance from the spinneret to the first takeup roll is from 16 to 20 feet,
- (d) cooling the spun yarn to less than 31°C before it
40 reaches the first takeup roll,

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5

(e) optionally, storing the spun yarn at a temperature of no more than 31°C,

10

(f) prior to the draw process, preconditioning the yarn under tension at a temperature of at least 60°C,

(g) drawing the yarn at a temperature of at least 60°C,

15

(h) optionally, allowing the drawn yarn to relax at a temperature of up to 190°C ,and

20

(i) crimping the drawn yarn at a temperature of 70 to 120°C and using a crimper volume of 10 to 50 percent more than the crimper volume of crimper used to make comparable PET.

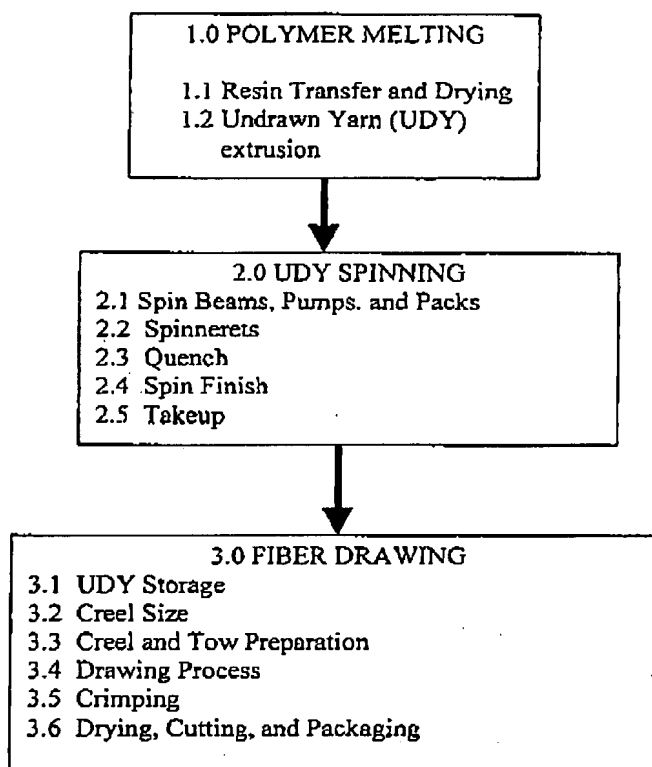
10. The process of any one of the preceding claims wherein step (g) involves at least two draws, the first carried out at least 60°C and the second and subsequent draws, if any, carried out at a higher temperature than the first up to the melting point of the yarn.

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Figure 1

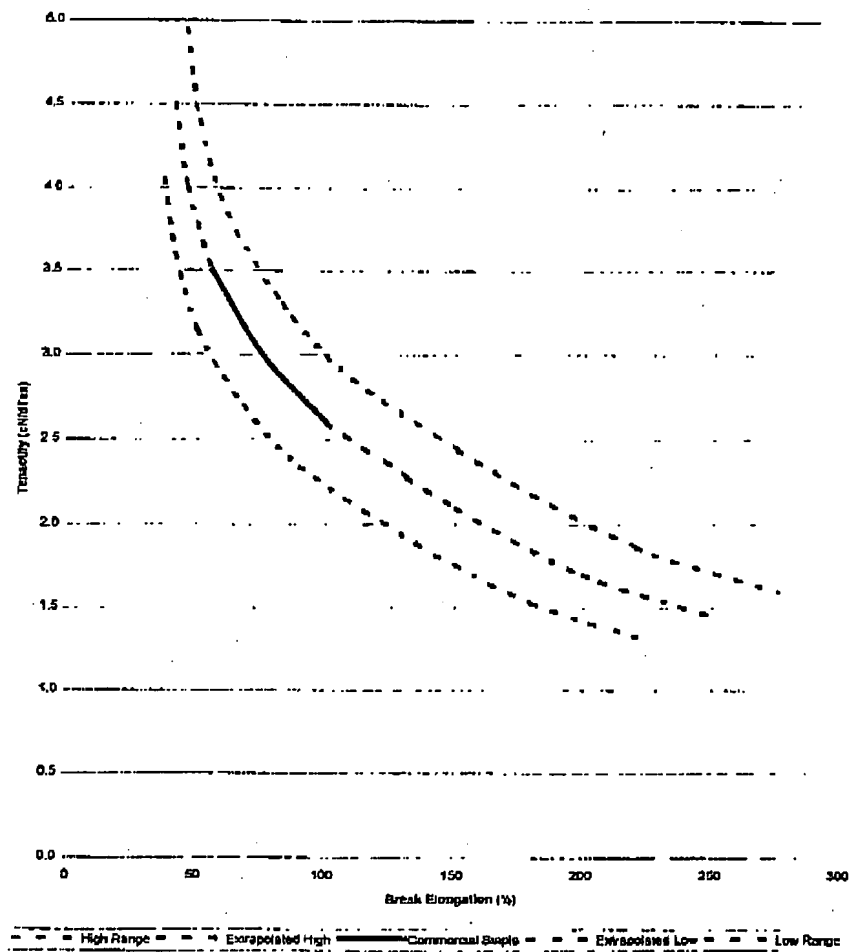


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Figure 2

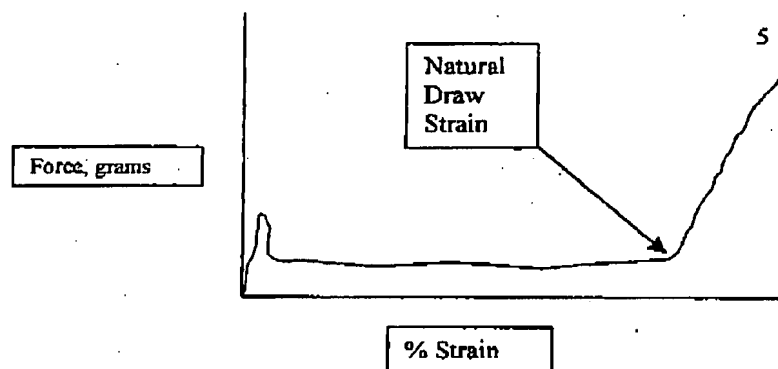


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Figure 3

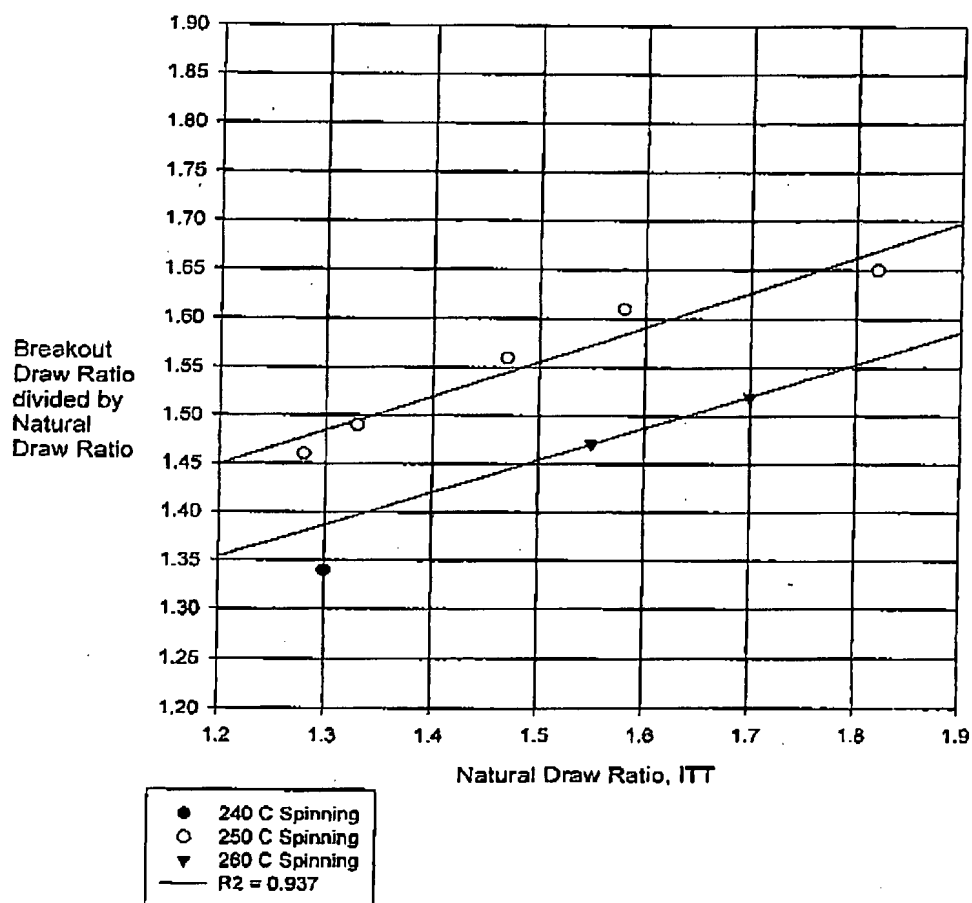


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Figure 4

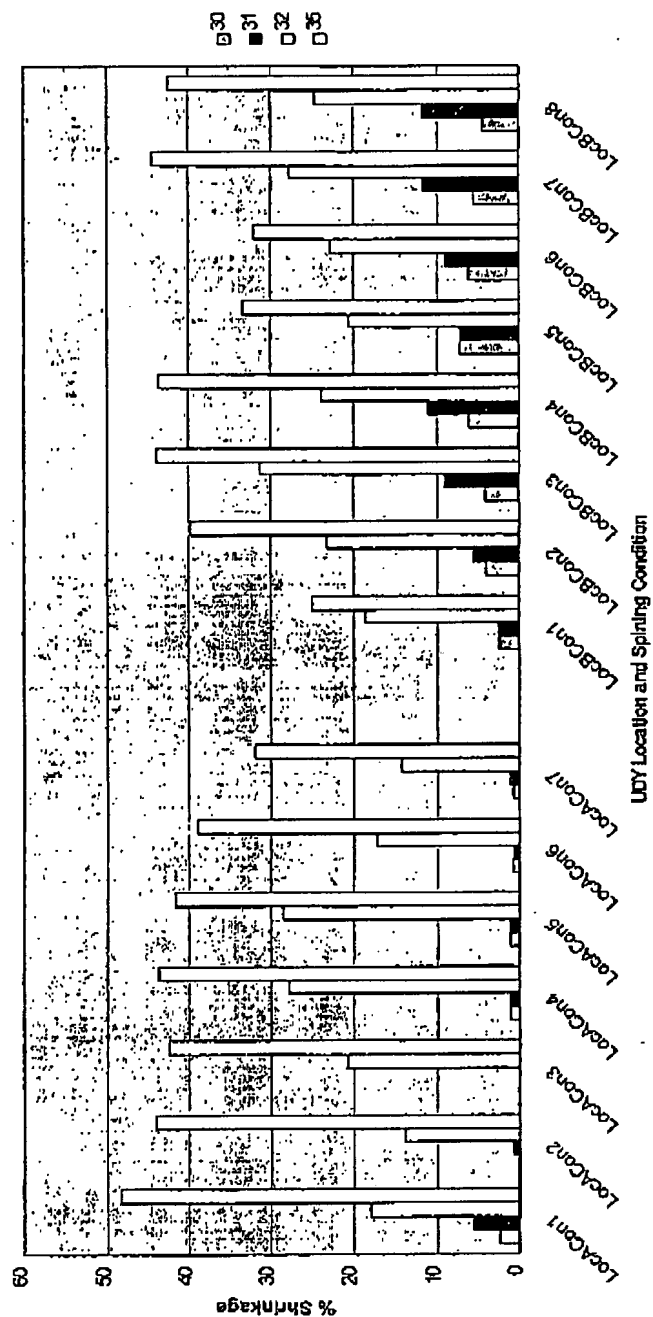


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Figure 5

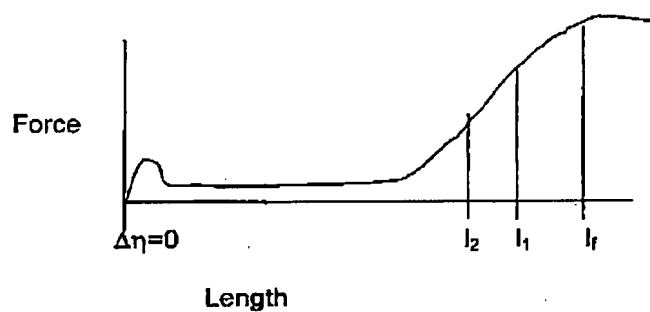


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Figure 6

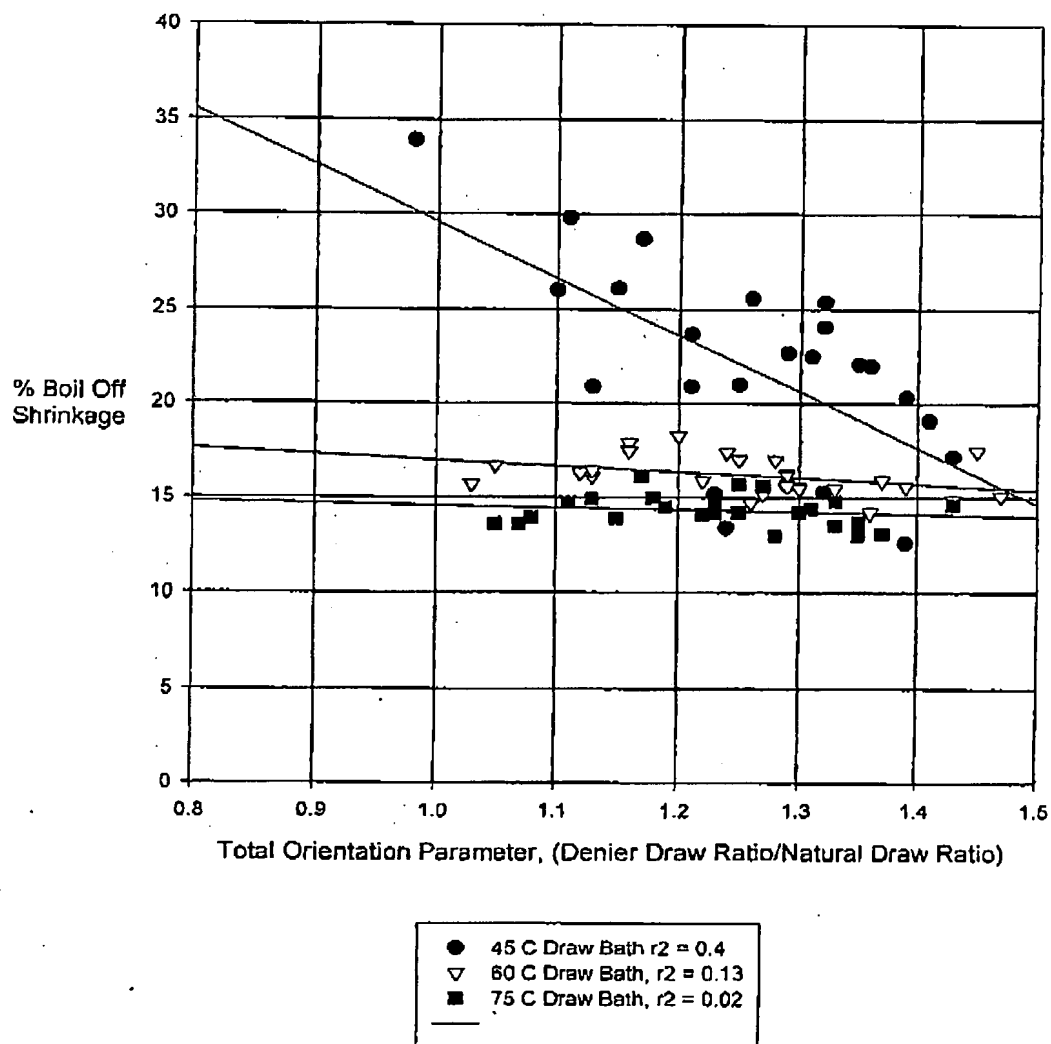


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Figure 7

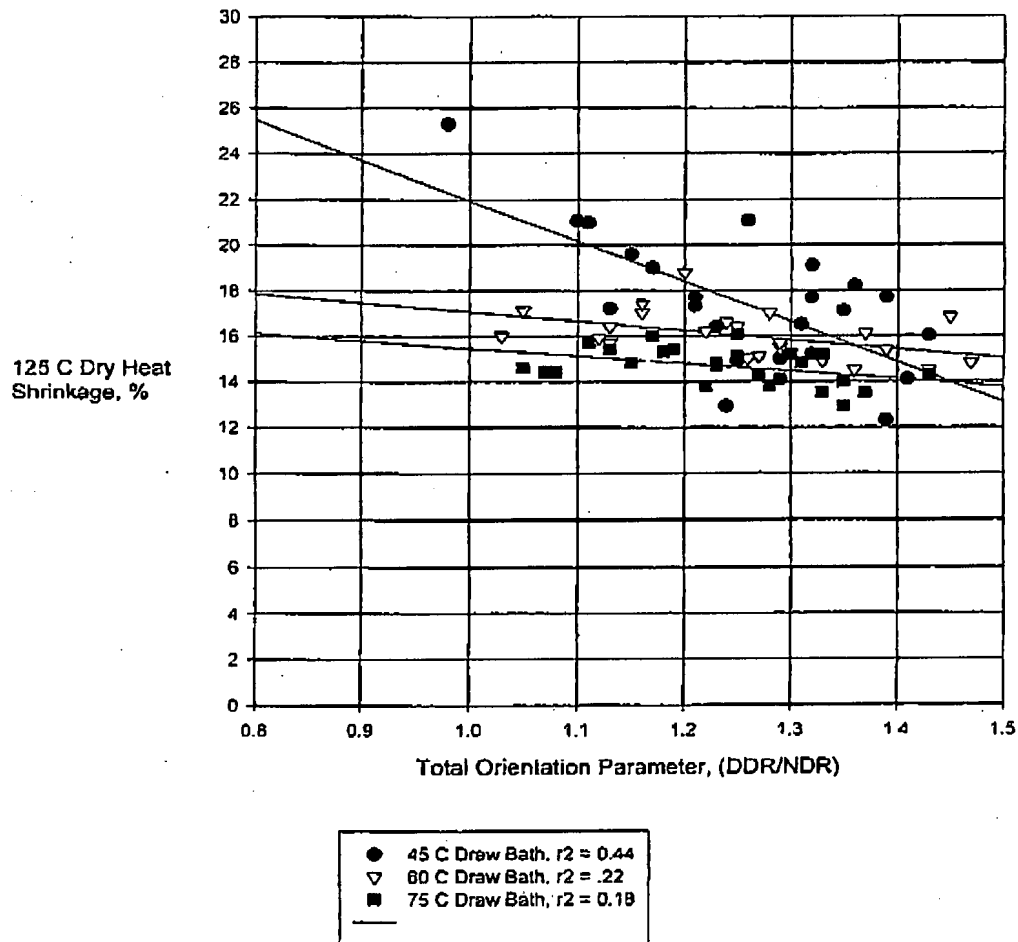


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Figure 8

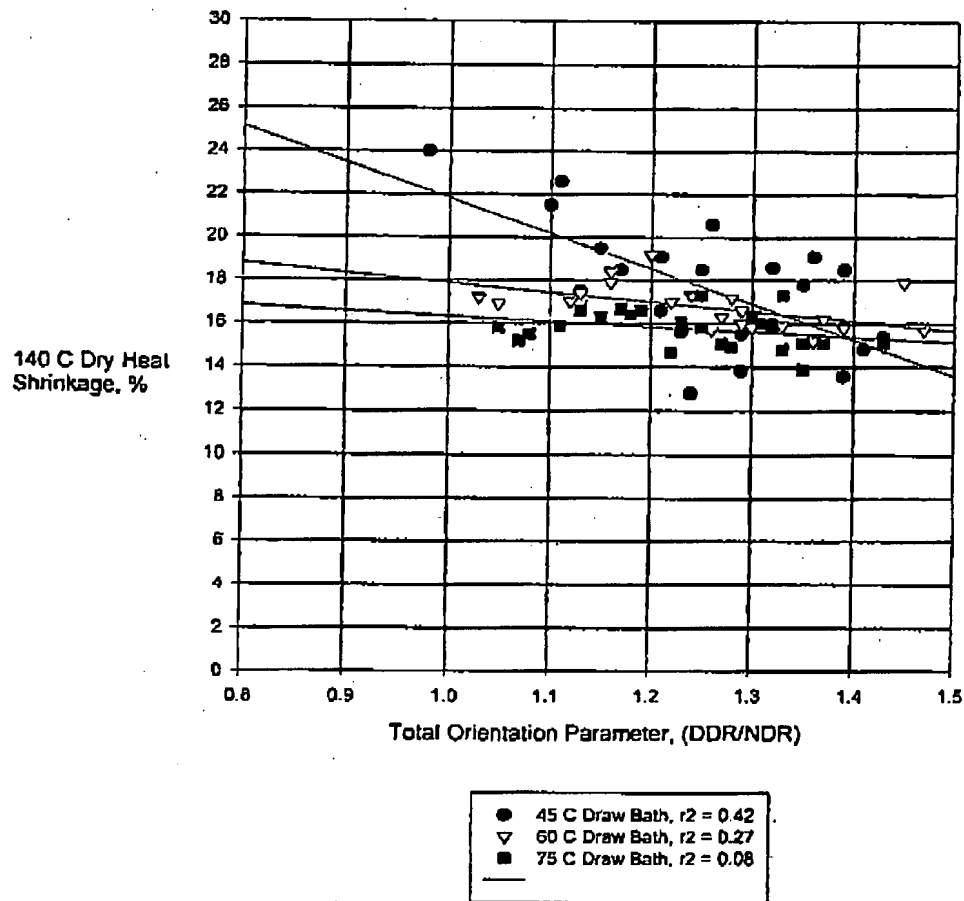


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Figure 9

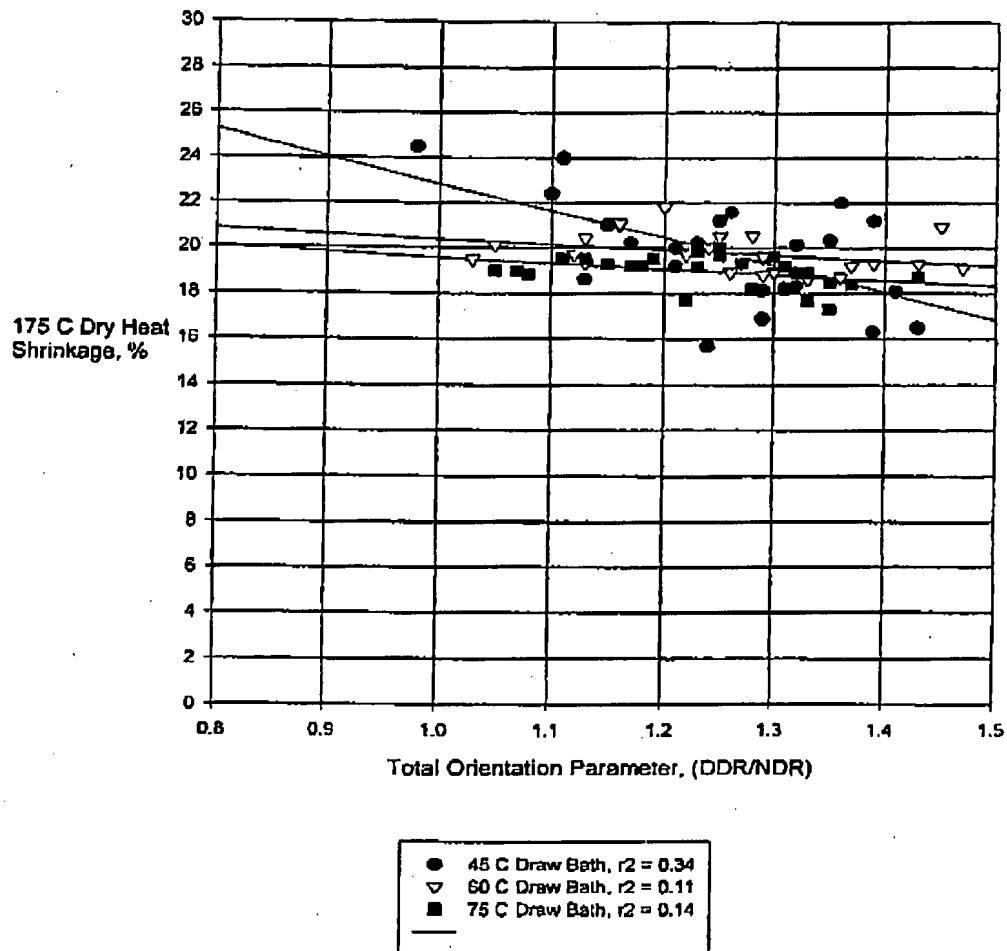


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Figure 10

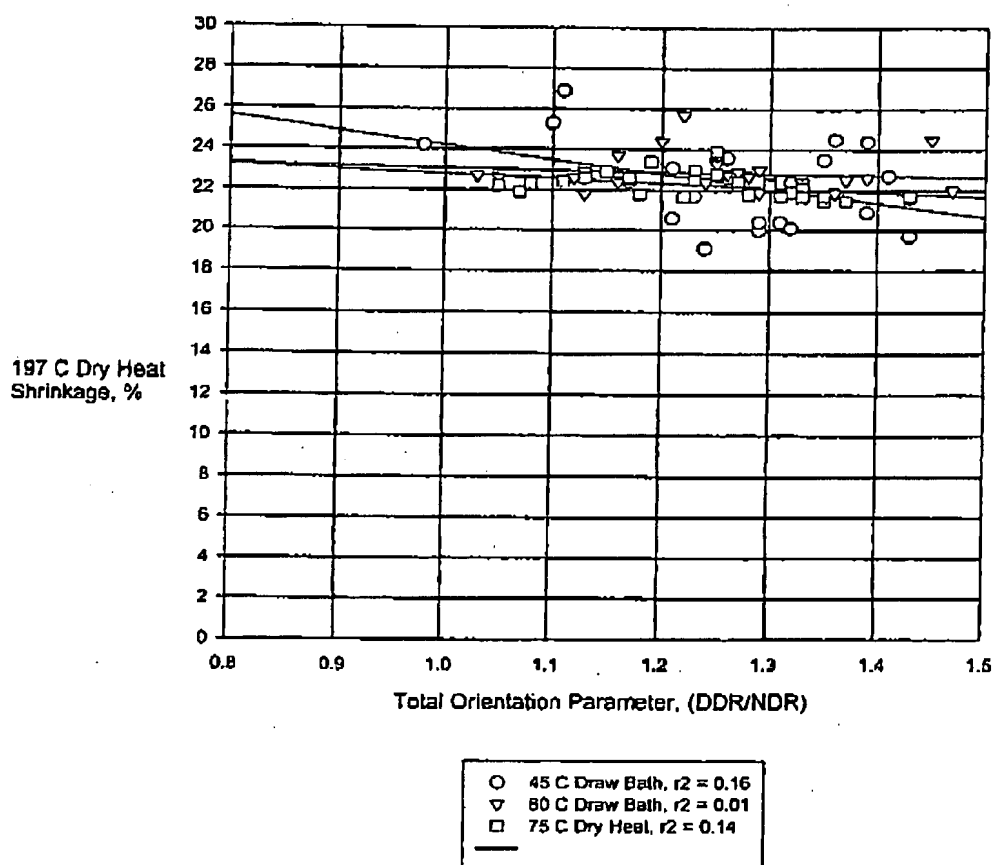


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Figure 11

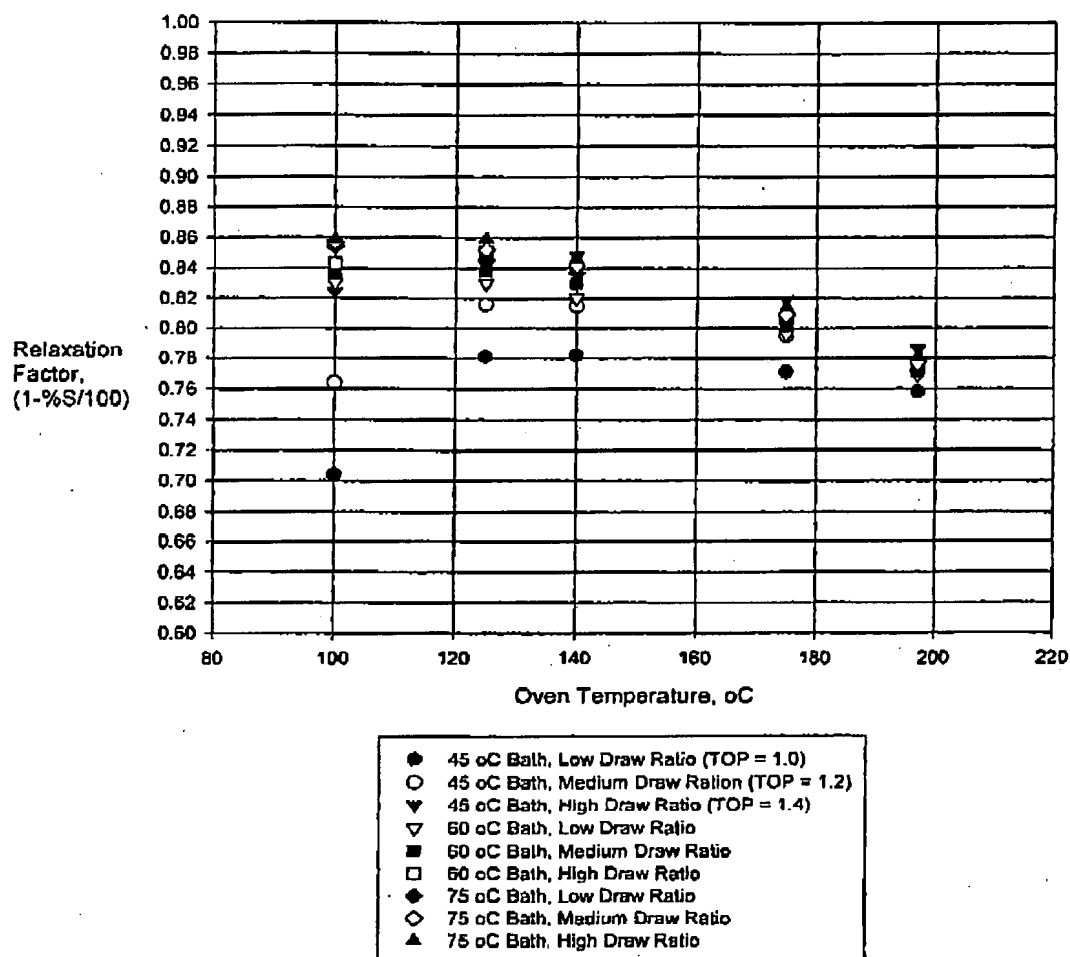


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Figure 12

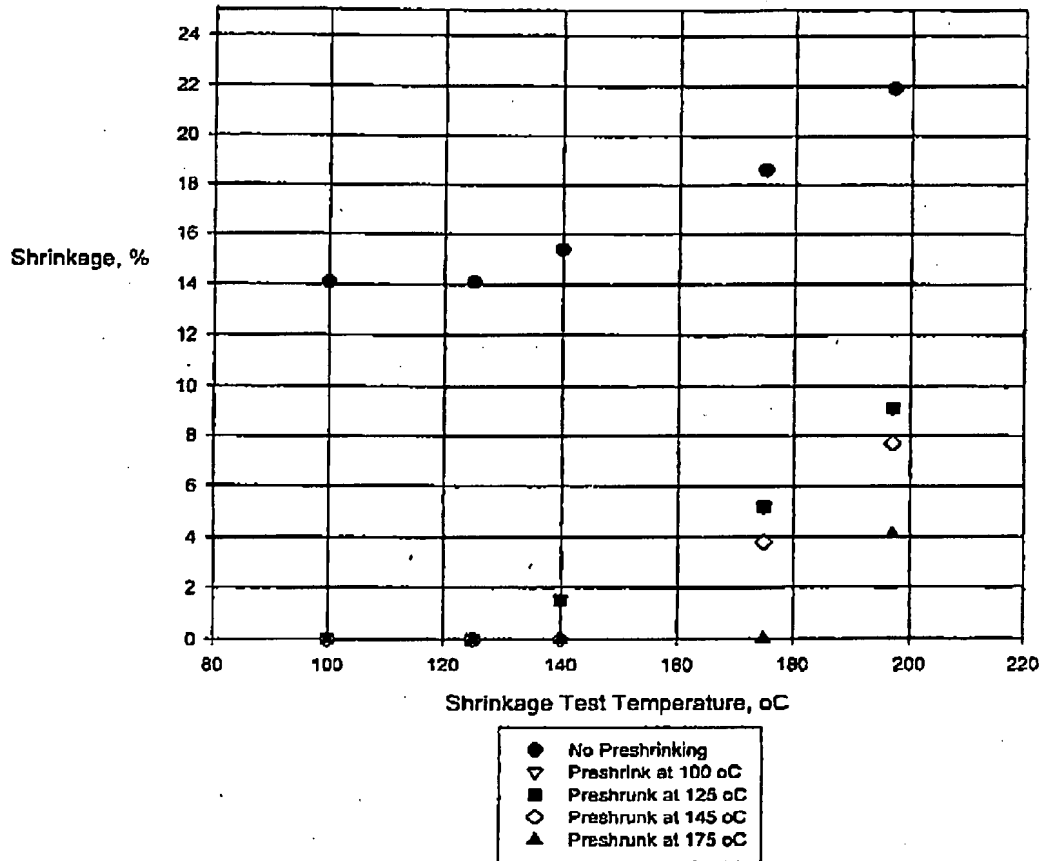


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Figure 13

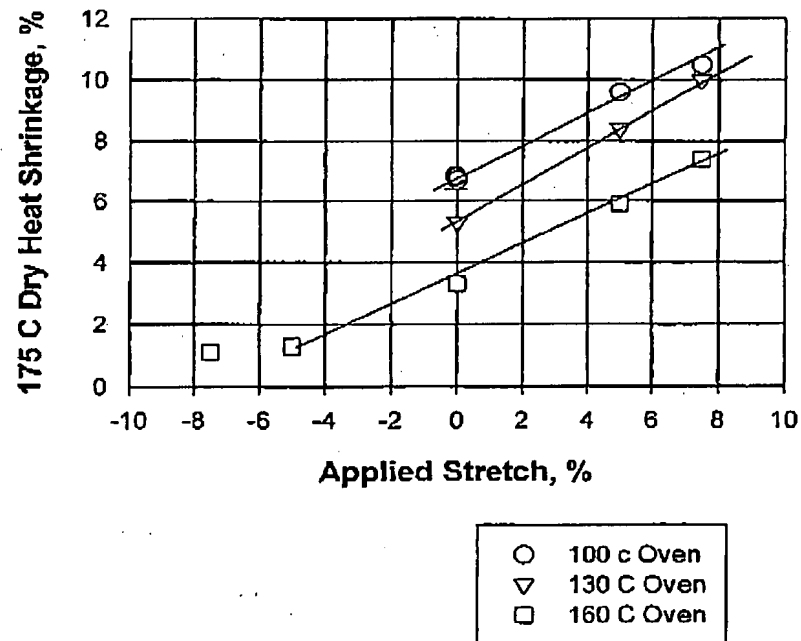


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Figure 14

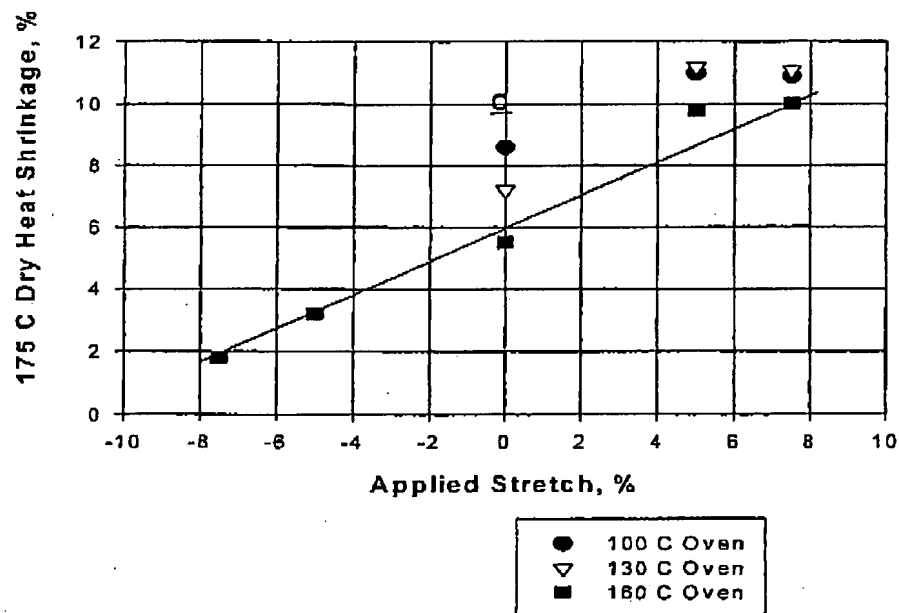


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Figure 15

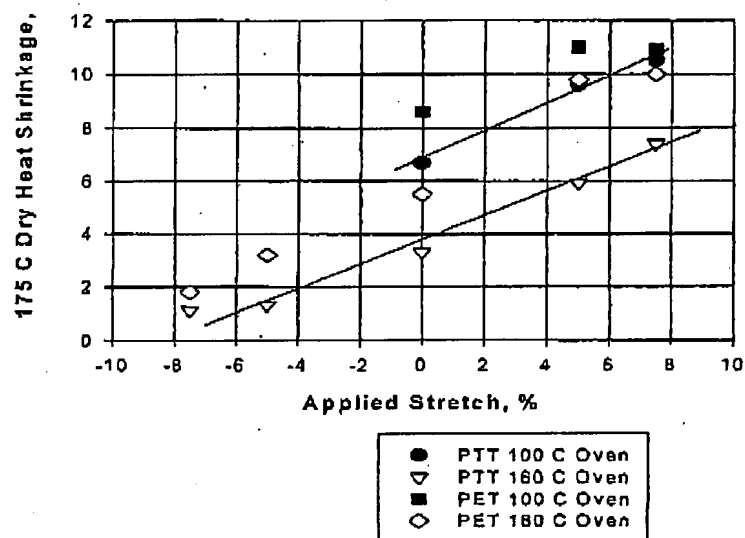


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Figure 16

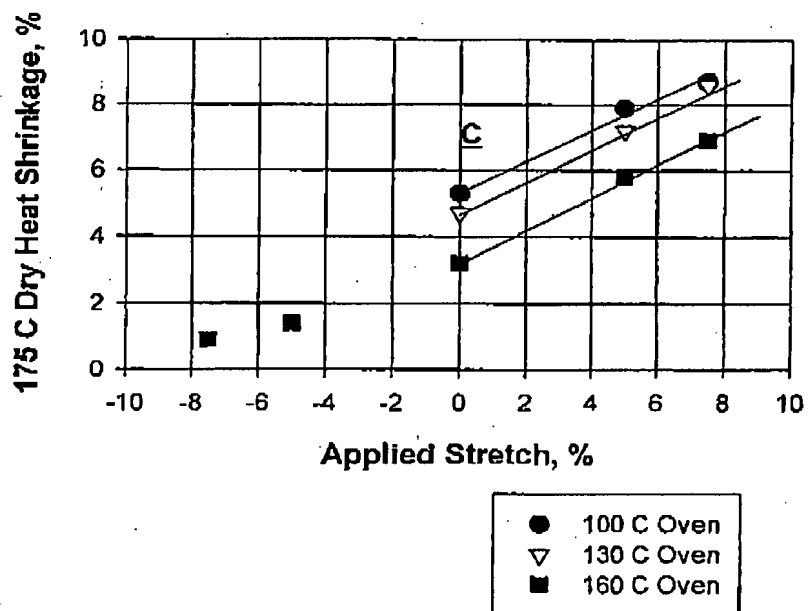


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Figure 17

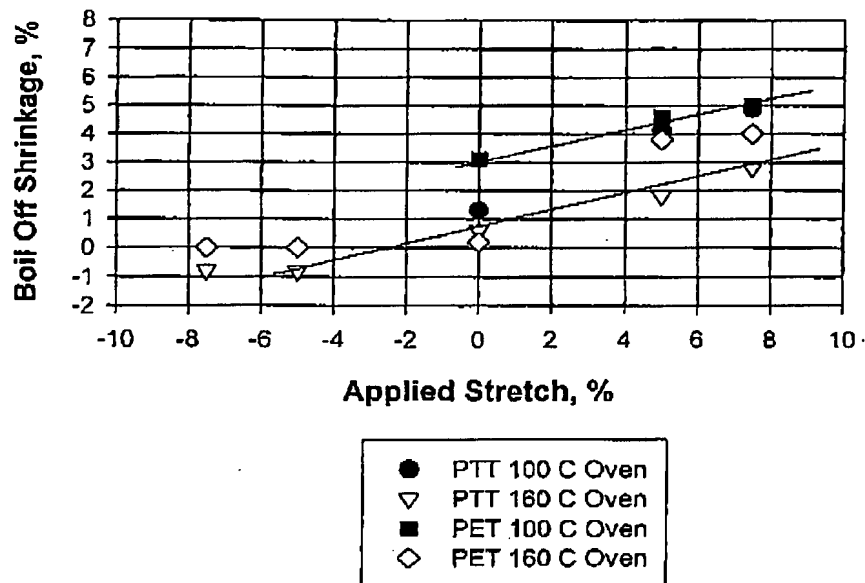


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Figure 18

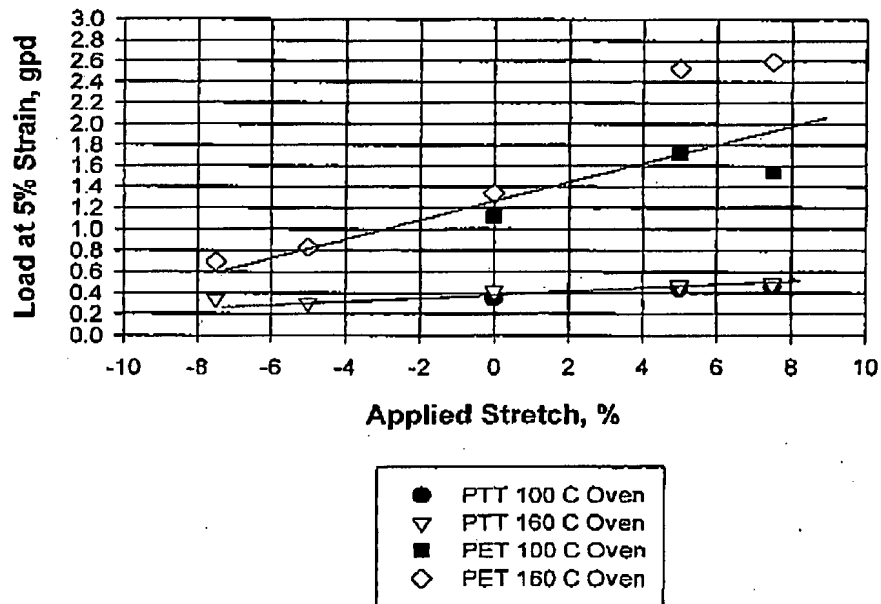


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Figure 19

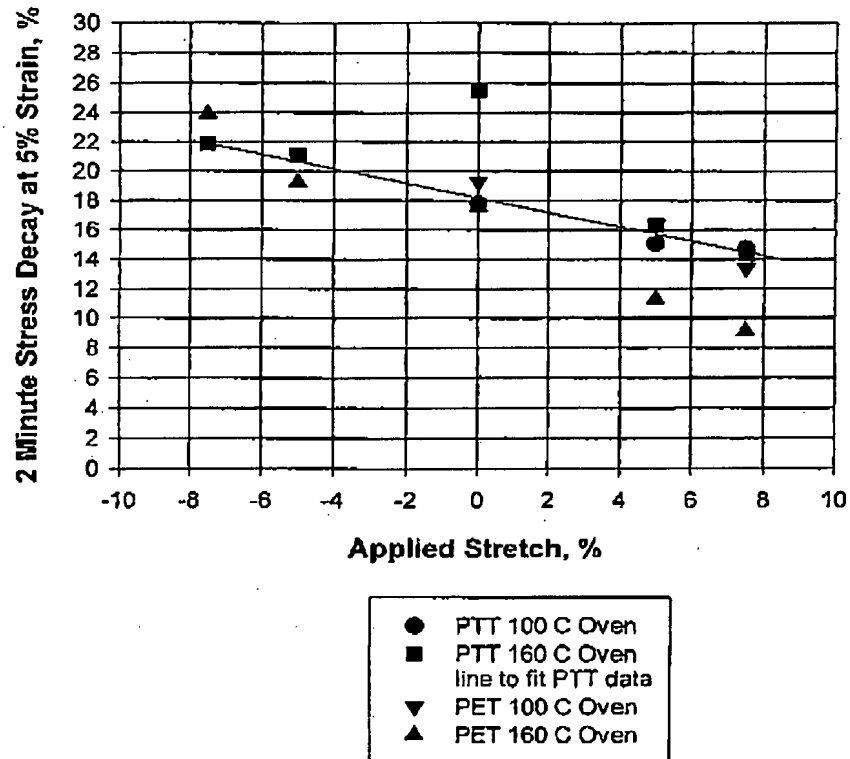


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Figure 20

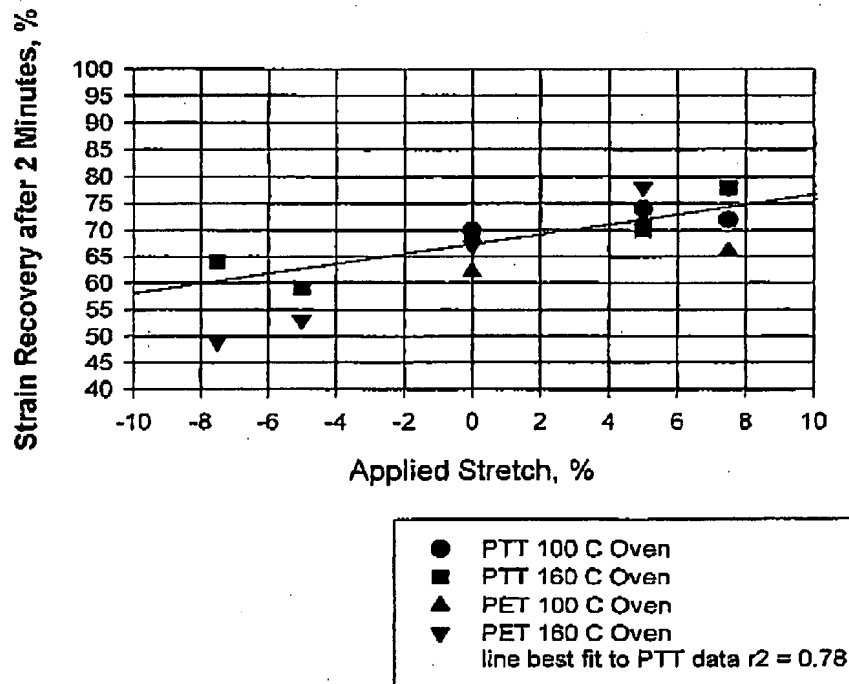


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Figure 21



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(19) World Intellectual Property Organization
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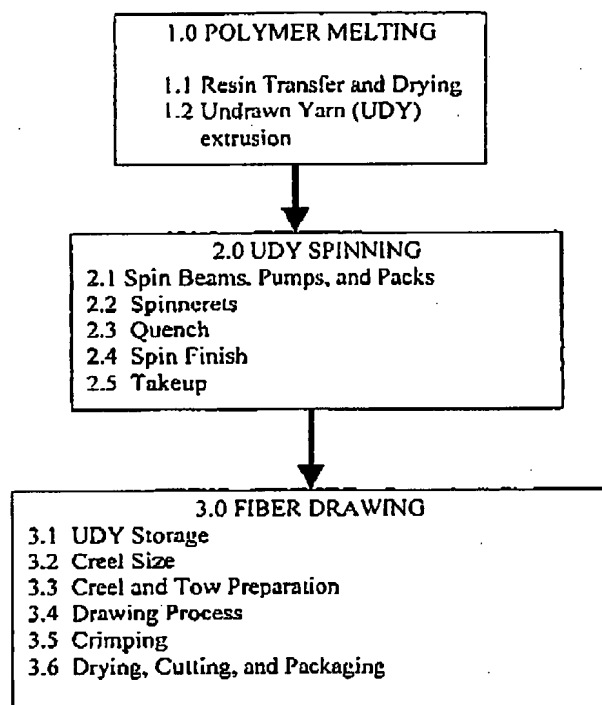
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60/189,531 15 March 2000 (15.03.2000) US(71) Applicant (for all designated States except US): **SHELL OIL COMPANY [US/US]; One Shell Plaza, P.O. Box 2463, Houston, TX 77252-2463 (US).**

(72) Inventors; and

(75) Inventors/Applicants (for US only): **CASEY, Paul.**

Karol [US/US]; 6126 Plantation Bay Drive, Katy, TX 77449 (US). DANGAYACH, Kailash [US/US]; 755 Windbreak Trail, Houston, TX 77079 (US). OLIVERI, Linda, Harvey [US/US]; 23042 Lanham Drive, Katy, TX 77450 (US). SHIFFLER, Donald, Albert [US/US]; 1700 Hardee Road, Kinston, NC 28504 (US).

(74) Agent: **HAAS, Donald, F.; Shell Oil Company, One Shell Plaza, P.O. Box 2463, Houston, TX 77252-2463 (US).**(81) Designated States (national): **AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CR, CU, CZ, DE, DK, DM, DZ, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW.**(84) Designated States (regional): **ARIPO patent (GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW), Eurasian***[Continued on next page]*(54) Title: **POLY(TRIMETHYLENE) TEREPHTHALATE TEXTILE STAPLE PRODUCTION**

(57) Abstract: A process for making textile staple fibre from poly(trimethylene) terephthalate (PTT) which comprises: (a) melt extruding PTT polymer at 245 to 253 °C, (b) spinning the extruded PTT into yarn using at least one spinneret, (c) moving the spun yarn to a first takeup roll wherein the distance from the spinneret to the roll is from 16 to 20 feet, (d) cooling the spun yarn to less than 31 °C before it reaches the roll, (e) prior to the draw process, preconditioning the yarn under tension at a temperature of at least 60 °C, (f) drawing the yarn at a temperature of at least 60 °C, (g) allowing the drawn yarn to relax at a temperature of up to 190 °C, and (h) crimping the drawn yarn at a temperature of 70 to 120 °C, and decreasing the drawn yarn feed denier into the crimper by 10 to 60 percent by denier.

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IT, LU, MC, NL, PT, SE, TR), OAPI patent (BF, BJ, CF,
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For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

INTERNATIONAL SEARCH REPORT

| A. CLASSIFICATION OF SUBJECT MATTER IPC 7 D01F6/62 | | International Application No. PCT/US 01/08230 |
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| According to International Patent Classification (IPC) or to both national classification and IPC | | |
| B. FIELDS SEARCHED Minimum documentation searched (classification system followed by classification symbols) IPC 7 D01F | | |
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| Electronic data base consulted during the international search (name of data base and, where practical, search terms used) EPO-Internal, PAJ, WPI Data | | |
| C. DOCUMENTS CONSIDERED TO BE RELEVANT | | |
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| <input type="checkbox"/> Further documents are listed in the continuation of box C. <input checked="" type="checkbox"/> Patent family members are listed in annex. | | |
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| Date of the actual completion of the international search 8 October 2001 | | Date of mailing of the international search report 15/10/2001 |
| Name and mailing address of the ISA European Patent Office, P.B. 5018 Patentplan 2 NL-2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 37 851 epo nl, Fax (+31-70) 340-3018 | | Authorized officer D'Souza, J |

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Information on patent family members

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APPENDIX C - RELATED PROCEEDINGS APPENDIX

Application Serial No. 10/733,998

NONE

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